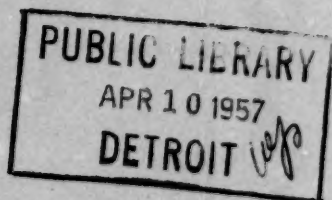*RR*

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

783. Liquid - liquid extraction procedures in inorganic analysis. [Groups IVA and IVB metals.] **[IV.]** T. S. West (Dept. Chem., Univ. of Birmingham). *Metallurgia, Manchr*, 1956, **53**, 234–236, 240.—Liquid - liquid solvent-extraction techniques for groups IVA (Ti, Zr, Hf, Th) and IVB (Sn, Pb, Ge) are reviewed.

[V.] A review of practical applications with particular reference to metallurgical analysis. T. S. West. *Ibid.*, 1956, **53**, 292–294.—The concluding section of this review covers the extraction of V, Nb, Ta, As, Sb and Bi. Examples of the techniques referred to include V in the quinquevalent form by the use of *iso*propyl ether, Ta (separation from Nb) in mixed acid solution by di*iso*propyl ketone, Nb in conc. HCl by a methyldioctylamine - xylene mixture, As in the tervalent form by a variety of solvents, Sb in the quinquevalent form in HCl (3 to 9 N) by diethyl or *iso*propyl ether and Bi as the carbamate complex by diethyl ether in the pH range 1 to 8.
S.C.I. ABSTR.
J. O. LAY

784. Ammonium aurintricarboxylate as a reagent in inorganic analysis. I. Colour reactions with metal ions. A. K. Mukherji and A. K. Dey (Chem. Dept., Univ. Allahabad, Allahabad, India). *Z. anal. Chem.*, 1956, **152** (6), 424–426.—Ammonium aurintricarboxylate (aluminon) gives characteristic colours or ppt. with many metal ions at concn. of 0·1 M. In particular, U and Th give very intense violet colours which obey Beer's law in the range of concn. 10^{-5} to 10^{-3} M.
A. R. ROGERS

785. The application of ferrocyanides in analytical chemistry. T. Gaspar y Arnal and J. Vega Bragado (Univ. Valladolid, Spain). *Chim. Anal.*, 1956, **38** (10), 343–352.—A quick method for the volumetric determination of K, which depends on the insolubility of the complex calcium potassium ferrocyanide, is described. Sodium ferrocyanide soln. is used as titrant and ammonium molybdate as an external indicator, and to effect complete pptn. the final soln. is arranged to contain ≮50% of ethanol. Ammonium ions react similarly and details of experiments to estimate NH_4^+ and K^+ when present together are given.
D. G. FORBES

786. Improvements in rapid qualitative analysis. A. Schleicher (Anal. Lab. der Rheinisch-Westfälischen Tech. Hochschule, Aachen). *Mikrochim. Acta*, 1956, (4–6), 663–666.—A brief report of a method of rapid qualitative analysis is given, by which a systematic analytical scheme is avoided, sensitive and specific reactions being used instead. Rapid qualitative and approximate quantitative results are obtained with small amounts of material.
M. F. C. LADD

787. Systematisation of qualitative microchemical analysis. M. E. Steimetz (Fac. Pharm., Univ. Nancy, France). *Chim. Anal.*, 1956, **38** (10), 365–366.—A system for the qual. analysis of mixtures of inorganic ions and of mixtures of organic compounds is described and is claimed to be exceedingly rapid. The reagents are divided into four groups, and the members of any one group have no action on each other. The sample soln. is treated successively with the reagents of each group, and the components are deduced from the reactions observed.
D. G. FORBES

788. Microchemical detection of several elements using ion-exchange resin. H. Kakihana. *Mikrochim. Acta*, 1956, (4–6), 682–688.—Colourless anion-exchange resins, impregnated if necessary, are used as media for the detection of Co^{II}, Cr^{VI} and Ge^{IV}, in approx. 1 N acid, with the reagents NH_4SCN, H_2O_2 and haematoxylin, respectively. The identification limits are 0·16 μg, 0·3 μg and 0·005 μg, and the concentration limits are 1 in 1·3 × 10^5, 1 in 1 × 10^5 and 1 in 6 × 10^6, respectively. The limitations imposed on these tests by the presence of other cations and of anions are considered.
M. F. C. LADD

789. Identification of minute amounts of metals and alloys by electrosolution and electrophoresis. C. L. Brown and P. L. Kirk (Sch. of Criminology, Univ. California, Berkeley, U.S.A.). *Mikrochim. Acta*, 1956, (11), 1593–1599 (in English).—A simple electrographic method is described for extracting micro quantities of material from the surface of metals or alloys, before electrophoretic separation and identification. Kojic acid - oxine (in visible and u.v. light), dithizone and Eriochrome black T are used as spot reagents to reveal the separated metals.
A. R. ROGERS

790. Methods of quantitative microchemical separations. P. E. Wenger and D. Monnier. *Mikrochim. Acta*, 1956, (4–6), 1075–1095.—At the 1st International Congress on Microchemistry, progress was reported in the following techniques of microchemical separations—(*i*) precipitation, (*ii*) complex formation, (*iii*) titration, (*iv*) distillation, (*v*) extraction, (*vi*) chromatography, (*vii*) electrolysis, (*viii*) polarography, (*ix*) spectrophotometry. (158 references.)
M. F. C. LADD

791. Photometric determination of indicator end-points. T. Higuchi, C. Rehm and C. Barnstein (Sch. Pharmacy, Univ. of Wisconsin, Madison, U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1506–1510.—A method is described for determining titrimetric indicator end-points photometrically by plotting the relative concn. of the acidic and basic forms of the indicator in solution against the vol. of standard acid or alkali solution added, or its reciprocal. The method can be applied to aqueous or non-aqueous systems and offers advantages over other similar methods in that the procedures described are not

dependent on data obtained at or near the end-point. The standard deviation ranges from 0·6% for four results on the titration of urea in acetic acid to 0·004% for five results on the standardisation of N NaOH against benzoic acid. K. A. PROCTOR

792. Spectrophotometric evaluation of universal indicators. F. Čůta and K. Kámen (Tech. High School for Chem., Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1956, **21** (4), 880–885 (in German).—The authors' universal indicator consisting of a mixture of bromothymol blue, trinitrobenzene, methyl orange, methyl red, pentamethoxyl red, phenolphthalein and o-cresolphthalein (cf. *Coll. Czech. Chem. Commun.*, 1936, **8**, 395) is compared spectrophotometrically with that of Kolthoff (*Pharm. Weekbl.*, 1929, **66**, 67) and Bogen (*Amer. Med. Ass.*, 1927, **89**, 199). Graphs showing extinction plotted against wavelength at various pH values, and extinction against pH at various wavelengths are given for each indicator. These show that the authors' indicator is more sensitive over the greater part of the pH scale. P. S. STROSS

793. Lophine, a new chemiluminescent indicator. L. Erdey and I. Buzás (Budapest Univ., Hungary). *Anal. Chim. Acta*, 1956, **15** (4), 322–324 (in English).—A 0·4% ethanolic or 1% acetonic soln. of 2:4:5-triphenyliminazole (lophine) can be used as a chemiluminescent indicator for the end-point of acid - base titrations, light being emitted in basic or oxidising media and in the presence of a catalyst. It has the same pH range and is as sensitive to CO_2 as phenolphthalein. The acid soln. is titrated with N or 0·1 N base and, in addition to 1 ml of lophine soln., should contain a small amount of 3% H_2O_2 and 5% $K_3Fe(CN)_6$ soln. The accuracy is within $\pm 0·4\%$ for titrations of N soln. W. J. BAKER

794. Micro-titrations with ethylenediaminetetra-acetic acid. XV. 1-(2-Pyridylazo)-2-naphthol as compleximetric indicator. H. Flaschka and H. Abdine (Inst. med. Chem. und Pregl-Lab., Univ. Graz, Austria). *Mikrochim. Acta*, 1956, (4–6), 770–777.—With PAN [1-(2-pyridylazo)-2-naphthol] as the indicator it is preferable to back-titrate an excess of EDTA with a standard copper solution, when the violet of the copper - PAN complex changes to yellow. No indicator correction is required. In acetic acid medium, Ni^{2+}, Co^{2+}, Zn^{2+}, Cd^{2+}, Cu^{2+}, Pb^{2+} and Fe^{2+} may be determined in the presence of alkaline earths. By titrating first in acid and secondly in ammoniacal medium, pairs of metals such as Ni - Mg, Zn - Mn and Fe - Ca may be determined in succession. M. F. C. LADD

795. Diethylenetetra-ammonium sulphatocerate as volumetric reagent. III. Ferrous ethylenediamine sulphate method. B. Singh, S. Singh and H. Singh (Panjab Univ. Coll., Hoshiarpur, India). *Anal. Chim. Acta*, 1956, **15** (4), 320–321 (in English).—Diethylenetetra-ammonium sulphatocerate (I) can be used as an oxidising agent in acid soln. for the indirect determination of H_2O_2, $Na_2C_2O_4$, KSCN, Cu_2Cl_2, ferrous ammonium sulphate and quinol. An excess of a 0·02 N soln. of I is added to the sample soln., which is then adjusted to between 1 and 2 N H_2SO_4, and the excess of I is titrated potentiometrically or volumetrically with 0·02 N ferrous ethylenediamine sulphate at 20°. About 10 ml of 3 N HCl should be added to the Cu_2Cl_2 soln. which, as well as those containing $Na_2C_2O_4$ and KSCN, must be heated and then cooled before titration. W. J. BAKER

796. Gasometric titrimetry. O. R. Gottlieb (Inst. Quím. Agricola, Rio de Janeiro). *Bol. Inst. Quím. Agric.*, 1955, No. 42, 88 pp.—A comprehensive review is given of the principles and applications of gasometric titrimetry, in which the end-point of a titration is determined from the change in volume or pressure of a gas evolved, either directly from the reactants or from an indicator. The determinations described include acid - alkali, oxidation - reduction, precipitation and complex-formation reactions. Results are comparable in accuracy to those of visual end-point titrations, but the titration needs to be rather slow. An automatic recording apparatus is described. L. A. O'NEILL

797. Ion exchange in micro-analysis and in preparative micro-scale work. E. Abrahamczik (Anal. Lab. Badischen Anilin- und Sodafabrik A.-G., Ludgwigshafen am Rhein). *Mikrochim. Acta*, 1956, (4–6), 651–662.—The micro-analytical applications of ion exchange are reviewed for batch and column processes. Apparatus is described and the requirements governing the choice of ion-exchange materials for various purposes are listed. The steps in the complete procedure, i.e., preparation of ion-exchange material, charging, washing, elution and regeneration, are enumerated. Details are given of exchange dialysis and electrodialytic purification. M. F. C. LADD

798. Systematic chromatographic micro-analyses of gases. J. Janák (Inst. für Erdölforschung in Brünn, C.S.R.). *Mikrochim. Acta*, 1956, (4–6), 1038–1049.—The literature of gas chromatography, up to 1955, is reviewed. A method of direct microanalysis is described and the idea of a gas-chromatographic spectrum is formulated. About 0·1 to 2 ml of gases may be separated and estimated. The chemical and physical similarity of gases is no drawback to the method; paraffins, cycloparaffins, olefins, acetylenes, halogens and the inert gases have been resolved. The relation between the position of gases in the spectrum and their molecular structure is discussed. M. F. C. LADD

799. The application of countercurrent principles in micro gas-analysis. K. R. Peters (Inst. Verfahrenstech. und Technol. der Brennstoffe, Tech. Hochschule, Vienna). *Mikrochim. Acta*, 1956, (4–6), 1023–1030.—Small amounts of components of flowing gas mixtures can be quantitatively absorbed if the gas is led through a "ten-drop tube" countercurrent-wise to the absorption liquid. Examples quoted are the determinations of CO_2 or SO_2 in air and the total sulphur content of furnace gases. M. F. C. LADD

800. New methods in gas analysis. I. Measurement of tension by microchemical methods. (Vapour space analysis.) E. Schulek, E. Pungor and J. Trompler (Inst. anorg. und anal. Chem. der L. Eötvös Univ., Budapest). *Mikrochim. Acta*, 1956, (4–6), 1005–1022.—The basic principles of gas analysis are reviewed. A measuring device is described which permits quantitative sampling of gases. The individual gases are determined on separate portions of material. The procedure is illustrated by analysis of methanol and ethanol. M. F. C. LADD

801. Titanic acid gel as an adsorption layer in potentiometric chromatography. B. Kamieński and I. Mazur. *Bull. Acad. Polon. Sci., Classe III*, 1955, **3**, 325–328 (in English).—Titanic acid gel is about twice as sensitive as silica gel when used as an

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adsorbent for potentiometric chromatography. Equal amounts of the two gels were used in the experiments, one drop of each being applied to the end of a glass rod electrode and dried. One drop of the silica gel contained $5 \cdot 7 \times 10^{-5}$ g of SiO_2, whereas one drop of the titanium gel contained $2 \cdot 8 \times 10^{-7}$ g of TiO_2.   CHEM. ABSTR.

802. The ring-oven method. H. Weisz (Inst. für anorg. und anal. Chemie, Tech. Hochschule, Vienna). *Mikrochim. Acta*, 1956, (4–6), 667–681.—The ring oven is described and illustrated. (*Cf. Anal. Abstr.*, 1954, **1**, 2603, 2604.) A glass apparatus is illustrated by which precipitation with gaseous reagents may be carried out.   M. F. C. LADD

803. Preparation of linear potato-starch fraction for quantitative colorimetric iodimetry. J. L. Lambert and S. C. Rhoads (Kansas State College, Manhattan, Kan., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1629–1630.—The linear potato-starch fraction (amylose) is extracted by suspending raw potato starch in water for 2 hr. at 57° to 60°. The extracted material is recrystallised three times by pptn. with *n*-pentanol, is dehydrated by treatments with methanol and *n*-pentanol and dried at room temp. The average yield is about 2 g of dry linear fraction for 50 g of raw potato starch.   G. P. COOK

804. Bibliography of publications dealing with the polarographic method in 1955. J. Heyrovský (Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1956, **21** (Suppl. I), 1–76.—This is a continuation of the bibliography given in the author's Complete Bibliography of Polarographic Publications from 1922 to 1950, published as Part II of the Proceedings of the First International Polarographic Congress in Prague, 1951, and successive publications (*Coll. Czech. Chem. Commun.*, 1951, **16**, 430–453; 1952, **17**, Suppl. I, 1–31; 1953, **18**, Suppl. I, 1–46; 1954, **19**, Suppl. I, 1–38; 1955, **20**, Suppl. I, 1–61).   N. E.

805. The contribution of radiochemistry to the theory of chemical analysis. T. Schönfeld (Chem. Lab., Univ., Vienna). *Mikrochim. Acta*, 1956, (4–6), 1050–1067.—Certain factors determine the suitability of radioactive materials for physical and chemical studies, *e.g.*, sensitivity, simplicity, selectivity and ability to distinguish between similar materials, one only of which is radioactive. The processes of analysis, precipitation, co-precipitation phenomena and ion exchange are considered in relation to radiochemical studies. M. F. C. LADD

806. Development of the electrographic method of analysis. R. A. Irkovskiǐ (Ostrava Mining Inst., Czechoslovakia). *Zavod. Lab.*, 1956, **22** (8), 898–904. —A review with 17 references.   G. S. SMITH

807. How to minimise errors in neutron activation analysis. R. C. Plumb and J. E. Lewis. *Nucleonics*, 1955, **13** (8), 42–46.—Errors in the actual measurement of the activity produced may arise as a result of incomplete chemical separation of the activity sought, production of given activities by transmutation of other elements present, neutron-flux variations in the region of the sample, and self-shielding within the sample. Possible methods of avoiding these sources of error are discussed.
PHYS. ABSTR.

808. Comparative evaluation of methods of sedimentary analysis. N. A. Figurovskiǐ and T. B. Gavrilova (Moscow State Univ.). *Zavod. Lab.*, 1956,

22 (8), 891–896.—Suspensions of quartz (very stable even at high concn. of the dispersed phase) and $BaSO_4$ (unstable at relatively low concn. of the dispersed phase) in water are subjected to sedimentary analysis by various known methods. It is considered that stable suspensions are examined preferably by methods that can be used with the lowest concn. of the dispersed phase, but otherwise any of the published methods are suitable. With unstable suspensions it is essential to use methods applicable to the minimum concn. ($\nprec 0 \cdot 3 \%$ by wt.) of the solid phase.   G. S. SMITH

2.—INORGANIC ANALYSIS

809. The determination of humidity in gases. J. Pech, S. Zedek and M. Wittová (Research Inst. Synth. Rubber, Gottwaldov, Czechoslovakia). *Chem. Průmysl*, 1955, **5** (5), 219.—The reaction—

$$Mg_3N_2 + 6H_2O = 3Mg(OH)_2 + 2NH_3$$
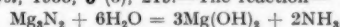
has been used for the determination of water, in particular of humidity in gases. The NH_3 is absorbed in $0 \cdot 1$ M H_3BO_3. The electrical conductivity of the resulting solution of ammonium salt is compared with that of the pure solution of H_3BO_3. The method was modified for progressive determinations.   J. ZÝKA

810. Separation of certain alkali-metal and alkaline-earth cations by electrochromatography. G. H. Evans and H. H. Strain (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1560–1563.—The electrochromatographic separation of the alkali metals from the alkaline earths varies greatly with the composition and pH of the background electrolytic solution. In most acidic solutions both groups migrate rapidly as cations with much cross-contamination of the zone. In weakly acidic and alkaline solutions of complex-forming acids (*e.g.*, EDTA), the alkaline earths form complexes which migrate as anions and separate completely from the uncomplexed alkali-metal cations. The most effective separation of Ba, Sr, Ca and Mg occurs when these ions are but partially complex. In acid solutions Ba migrates the fastest, followed by Sr, Ca and Mg, but in weakly acid and alkaline solutions Mg migrates the fastest, followed by Ca, Sr and Ba. The separation of the alkali-metal ions is unaffected by variations in the background electrolytic solution. Under all the conditions tested, Cs, Rb and K form a single rapidly migrating zone, followed by Na and Li. No simple relationship is observable between the electrochromatographic sequence of the alkali-metal ions and the sequences observed in columns or on paper. K. A. PROCTOR

811. Analysis for industry [further applications of sodium tetraphenylboron for various determinations]. A. Sykes. *Ind. Chem. Mfr*, 1956, **32**, 164–166; 223–225.—A review is given of the use of Na tetraphenylboron for the determination of K, and of potassium, ammonium and organic bases; in titrations in non-aq. media; and the stability of its solutions and various qual. applications. (28 references.)   S.C.I. ABSTR.

812. A contribution to the analytical determination of lithium. J. Gottfried (Research Inst. Inorg. Chem., Ústí nad Labem, Czechoslovakia). *Chem. Průmysl*, 1955, **5** (9), 390–391.—Lithium can be

pptd. by means of potassium metaperiodate in the form of an insol. compound; the content of periodate can be determined iodimetrically. J. ZÝKA

813. Spectrophotometric determination of lithium. P. F. Thomason (Anal. Chem. Div., Oak Ridge Nat. Lab., Oak Ridge, Tenn., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1527–1530.—Lithium forms a coloured complex, intensified by acetone, with thoron in alkaline solution. For the determination in aqueous solution (10 to 80 μg per 10-ml final volume), add KOH soln. (20%) (0·2 ml) to an aliquot (1 ml) and dilute to 8 ml with water. Add thoron reagent (0·2% solution) (1 ml), dilute to 10 ml and measure the extinction at 458 mμ after 30 min. against a reagent blank. For the determination of concentrations of 1 to 10 μg per 10-ml final volume, proceed as described above, but add 7 ml of acetone instead of water after the addition of the KOH soln. Measure the extinction at 486 mμ against a reagent blank. The amount of Li is found by reference to a calibration curve. The colour is stable for at least 1 hr. There is no serious interference from Ca^{2+} and Mg^{2+} in an amount 10 times, and Na^+ 50 times, that of Li present. The accuracy is within ±3%. D. G. LLOYD

814. Determination of elemental carbon in sodium. L. P. Pepkowitz and J. T. Porter, II (Knolls Atomic Power Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1606–1607.—The method was devised to measure the elementary carbon content ($\not> 0.01\%$ by wt.) of sodium, used as the coolant in a stainless-steel heat-exchange system, since embrittlement of the steel due to grain boundary carburisation can occur. The sodium is converted into a salt and the carbon is then oxidised to CO_2, which is estimated by a freeze-out technique. *Procedure*—Break the glass tube or cup containing the sample, cut up the sodium, and dissolve it in a small vol. of water under N in a combustion tube. After neutralisation with 5 N H_2SO_4, add 10% in excess, and evaporate almost to dryness. The combustion tube, in the form of a test-tube, fits into a socket carrying an inlet tube for O, a delivery tube from the Van Slyke reservoir and an outlet tube to the measuring system, equipped with a three-way stopcock permitting the flushing of the tube with O before connection with the measuring system. Pass purified O through the combustion tube to remove N and air, stop the flow and connect to a gasometer. Add Van Slyke oxidising fluid (*J. Biol. Chem.*, 1940, **136**, 509) and heat at incipient boiling for 5 min. Sweep the gaseous combustion products into the gasometer with O and estimate by the freeze-out technique of Pepkowitz and Moak (*Anal. Chem.*, 1954, **26**, 1022). Run a blank to allow for C in the fuming H_2SO_4 in the oxidising fluid. By measuring the length or number of drops of Na in the sample tube its weight may be calculated to within ±10%. The standard deviation is ±0·001% for concn. of C of 0·01% or less. D. G. LLOYD

815. Spectrophotometric determination of potassium with sodium tetraphenylboron. R. T. Pflaum and L. C. Howick (Dept. of Chemistry, State Univ. of Iowa, Iowa City, U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1542–1544.—Ultra-violet spectrophotometry may be used in conjunction with existing methods for the determination of K^+ with Na tetraphenylboron (**I**). *Procedure*—Prepare an aq. soln. of sample at pH 4·0 to 5·0 and a filtered stock solution of **I** [1 g of reagent and 0·5 g of $AlCl_3.6H_2O$

or $Al(NO_3)_3.6H_2O$ in 100 ml of water]. Add reagent (5 ml) to sample (5 ml) soln. in a 15-ml graduated centrifuge tube and centrifuge for 3 min. Remove the supernatant liquid, wash the ppt. with a cold saturated soln. of the potassium salt (2×3 ml), leaving 0·5 ml of liquid with the ppt. Dissolve the ppt. in 5 ml of methyl cyanide - water (75:25), make the soln. up to 25 ml in a volumetric flask and determine the concn. of K^+ from the extinction at 266 mμ, against a blank, and a calibration curve. Absorption max. for the tetraphenylboron ion occur at 266 mμ and 274 mμ. Interference is caused by Tl^+, Ag^+, Hg^{2+}, NH_4^+, Cs, Rh and some amines; up to 1000 p.p.m. of Co^{2+}, Cu^{2+}, Fe^{3+} and Ni^{2+} is permissible and up to 2000 p.p.m. of Al^{3+}, Ca^{2+}, Li^+, Mg^+, Na^+, acetate, halides, NO_3^- and SO_4^{2-}. The method is valid over the range 5×10^{-5} to $7·5 \times 10^{-4} M$. The accuracy is within ±2%. D. G. LLOYD

816. Potassium determination with the X-ray spectrograph. L. B. Gulbransen (Mechanical Engng Dept., Washington Univ., St. Louis, Mo., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1632–1634.—An X-ray spectrographic procedure is described in which potassium K radiation is used for the quantitative analysis of concentrates and tailings. In constructing working curves, two series of standards ranging from 0·8 to 18·2% of K_2O and 57·8 to 61·5% of K_2O were used. No background correction was necessary. Results by this method agree to within 0·4% of K_2O of those obtained by chloroplatinate chemical analyses. After the samples have been ground to 200 mesh, the time for analysis varies from 3 to 6 min. K. A. PROCTOR

817. Iodimetric determination of copper. I. D. M. Kemp and E. F. C. H. Rohwer (Univ. of Stellenbosch, S. Africa). *J. S. Afr. Chem. Inst.*, 1956, **9** (1), 23–29.—A titration procedure is described in which KSCN is used to economise in KI in the titration of $CuSO_4$ solutions. The interference of As^V in the determination can be eliminated by adding 10% aq. $ZrCl_4$ solution (2·5 ml for each 0·1 g of As^V present). S.C.I. ABSTR.

818. Ultra-micro method for the direct determination of copper after chromatography on paper. A. Lacourt and P. Heyndryckx (Microchem. Dept., Univ. Brussels, Belgium). *Mikrochim. Acta*, 1956, (11), 1621–1648.—Titrimetric, photometric and spectrophotometric methods for the determination of ultra-micro quantities of Cu on paper before or after chromatography are compared. The reproducibility is improved by the chromatographic development. The lower limit of sensitivity by titration with 0·01 M EDTA is 3 μg of Cu. The sensitivity is improved to 0·1 μg by a photometric procedure which involves measurement of the intensity of the colour of the complex with rubeanic acid. With white light, the "paper blank" is large and variable, giving rise to errors; spectrophotometric determination with monochromatic light of 660 mμ reduces the blank and gives a precision of 1 to 0·1% for 0·1 to 3·0-μg quantities of Cu. A. R. ROGERS

819. Spectrographic analysis of cathode copper for metallic impurities. V. L. Ginzburg, I. N. Gramenitskiĭ and D. M. Livshits (Norilsk Mining Metallurgy Combine). *Zavod. Lab.*, 1956, **22** (8), 927–930. —Fusion at 1100° for 5 min. of drillings from copper containing < 0·03 to 0·05% of Ni, Fe, Sb, Sn, As, Bi and Zn does not affect the concn. of the impurities. For the spectrographic analysis of

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copper, electrodes are prepared by melting drillings in a graphite mould and excited in a Sventitskiï-type a.c. arc. G. S. SMITH

820. Spectro-analytical studies on ancient copper finds. J. Derkosch, F. X. Mayer and H. Neuninger (II Chem. Inst., Univ. Vienna). *Mikrochim. Acta*, 1956, (11), 1649–1661.—The composition of a number of copper objects of the late stone age and the early bronze age, together with ores from sites that were worked out in prehistoric times, has been investigated by emission spectrography. The difficulty of ascribing these objects to particular ore sites is considerable. A. R. ROGERS

821. 3-Hydroxy-1:3-diphenyltriazine as a reagent for copper(II), palladium(II) and nickel(II), and 3-hydroxy-1-p-chlorophenyl-3-phenyltriazine for titanium (IV). N. C. Sogani and S. C. Bhattacharyya (Gov. College, Ajmer, India). *Anal. Chem.*, 1956, **28** (10), 1616–1618.—The preparation and use of 3-hydroxy-1:3-diphenyltriazine (**I**) for the estimation of Pd^{II} and its separation from various elements, together with an indication of its high selectivity towards Cu^{II}, has been previously described (*cf. Anal. Chem.*, 1956, **28**, 81). The reagent is superior to salicylaldoxime and benzoin α-oxime in having a lower conversion factor (0·1303) and in being readily hydrolysed into water-soluble products. For the determination of Cu^{II}, which forms the complex $(C_{12}H_{10}N_3O)_2Cu$, optimum results are given at pH 2·3 to 3, HCl (N) and 10% (w/v) soln. of Na acetate and Na K tartrate being used for buffering, and the reagent being added as a 1% soln. (w/v) in 95% ethanol. Heating the solution hydrolyses any excess of reagent. The accuracy is within $\pm 0.2\%$. The same precautions as for Pd^{II} are taken in the separation of Cu^{II} from Ce^{IV}, Zr^{IV}, Fe^{II} and Fe^{III}. In the presence of Pb^{II}, acetic acid or HNO_3 is used instead of HCl to avoid pptn. of $PbCl_2$. For separations from Ti^{IV}, Sn^{IV} and molybdate, NaF or KF is added as masking agent. Separation of Cu^{II} from Pd^{II}, V^{IV} and vanadate cannot be effected owing to co-pptn. Interference is caused by Ag^+, Au^{3+}, Os^{4+} and CN^-. The separation of Pd^{II} from Rh^{III}, Ru^{III}, Ir^{III}, Th^{IV}, Sn^{IV}, Pb^{II}, Ti^{IV}, molybdate, tungstate, fluoride and phosphate may be effected by a procedure similar to that for Cu^{II}, with the same elements interfering. The accuracy is within $\pm 0.1\%$. Bivalent Ni may be estimated in the presence of Zn^{2+}, Mn^{2+}, Cd^{2+}, Mg^{2+}, As^{3+}, Be^{2+}, $PO_4{}^{3-}$ and F^- as a weighable ppt. at pH 5 to 5·5, although the specificity of **I** to it is not high. Sodium acetate is used as buffering agent, the excess of reagent being removed with 20% alcohol. For estimations of Ti^{IV}, 1-p-chlorophenyl-3-hydroxy-3-phenyltriazine may be used; this reagent is prepared analogously to **I**, but with p-chloroaniline instead of aniline. A soln. of the reagent [1% (w/v) in hot methanol] is added to the test soln. buffered to pH 2·3 with Na acetate and HCl. The ppt., after being boiled to remove methanol, is filtered off, washed, dried and ignited to TiO_2 for weighing. Quadrivalent Ti cannot be estimated in the presence of Zr^{4+}, ZrO^{2+}, Tl^{2+}, Ce^{4+}, Cu^{2+}, Pd^{2+}, Fe^{2+}, Fe^{3+}, molybdate and vanadate because of co-pptn., and interference is also caused by tungstate, phosphate and F^-. The accuracy is within $\pm 0.2\%$. D. G. LLOYD

822. Compleximetric titrations (chelatometry). XVII. The determination of copper, iron, aluminium and titanium. Z. Šír and R. Přibil (Res. Inst. for Powder Metall., Vestec, near Prague). *Coll. Czech.*

Chem. Commun., 1956, **21** (4), 866–872 (in German). —A method for the determination of Fe, Al and Ti in pyridine soln., by back-titration with $ZnSO_4$, with Eriochrome black T as indicator, is described. If NH_4F is added, Fe only is determined in the presence of Al and Ti. Iron and Cu give a deep-blue colour with catechol violet (**I**) in pyridine acetate buffer (pH 5·5 to 6·0), which is suitable for the detection of the end-point in EDTA titrations. Since Cu and Fe do not form complexes with F^- they can be determined in the presence of Al and Ti. The following methods are recommended for the analysis of Fe - Al - Cu alloys. Dissolve 1 g of alloy in HBr (5 ml), add KCl (2 g) and evaporate to dryness on a water bath. Dissolve the residue in dilute HCl $(1 + 1)$ (1 ml) and water (100 ml). Precipitate the Cu with H_2S; filter and ash the pptd. CuS, dissolve the residue in HCl (2 ml), remove any co-pptd. Fe and Al by pptg. the hydroxides with pyridine, and determine the Cu by titration with EDTA, with **I** as indicator. Combine the Fe and Al portions, boil to expel H_2S and dilute to 500 ml. To an aliquot (25 ml) add 0·05 M EDTA (30 ml), pyridine (5 ml) and **I** (10 drops) and back-titrate with $CuSO_4$ soln. To a further aliquot add NH_4F soln. (2%) (15 ml), EDTA (10 ml) and pyridine (10 ml), and back-titrate with $CuSO_4$ soln. The content of Fe is given by the second titration and the sum of Fe and Al is given by the first titration, Al being calculated from the difference. An alternative procedure involves pptn. of the Cu with oxine. Most metals, except Mg and the alkaline earths, interfere. P. S. STROSS

823. Increase in the specificity of the dithizone method of determining silver by the use of Complexone III [EDTA, disodium salt]. V. G. Goryushina and E. Ya. Gallis. *Zavod. Lab.*, 1956, **22** (8), 905–907.—Silver can be determined by extraction titration with dithizone (0·005% soln. in CCl_4) at pH 4·7 in the presence of EDTA (disodium salt), which forms complexes with Cu, Bi and Pb and prevents their interference even when the ratio to Ag is 100,000 to 1. Interference of Au ($\geqslant 20$ times that of Ag) is also prevented if the soln. is first boiled for 2 to 3 min. to give metallic gold through the reducing action of EDTA. G. S. SMITH

824. Analytical separation of beryllium from aluminium and iron. V. Ruml. *Chem. Průmysl*, 1955 **5** (11), 480–482.—By using a saturated solution of KF or NaF, Al^{3+} and Fe^{3+} in slightly acid solutions can be pptd. and removed as insol. fluorides, whilst Be^{2+} remain in the soln. Amounts of 5 to 50 mg of Be can be determined with an accuracy within $\pm 0.40\%$. This method is rapid and suitable for the direct determination of Be in minerals. J. ZÝKA

825. Rapid procedure for determination of magnesium and calcium in sodium chloride solution for electrolysis. Sinsaku Matui, Tadao Ueda and Sigeru Katumata (Developing Dept., Asahi Denka Kogyo Co., Arakawa-ku, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (6), 588–591.—In the presence of a large amount of Na, the titration of Ca or Mg with EDTA (disodium salt) gives high values, the error increasing linearly with increase in the amount of Na. The analysis of Mg and Ca (total 0·2 to 0·4%) in crude NaCl can, however, be satisfactorily carried out with Eriochrome black T as indicator for total Ca and Mg and murexide (0·2 g in 2 ml of 2 N NaOH) as indicator for Ca by the addition of Mg (0·02 N, 5 ml), which makes the

end-point clearer. Magnesium may also be colori-metrically determined with Titan yellow in $0.1 N$ NaOH at 535 mμ. The Ca is calculated from the difference. The presence of Fe^{2+} (> 20 mg per litre), Al (> 20 mg), Hg (> 10 mg) and Cu (> 20 mg) vitiates the method.　　　　K. SAITO

826. Micro-gravimetric and spectrophotometric determination of calcium by means of loretin. J. Gillis, J. Van Der Stock and J. Hoste (Univ. Ghent, Belgium). *Mikrochim. Acta*, 1956, (4–6), 760–769. —Semi-micro and micro-gravimetric methods for the determination of Ca by loretin (8-hydroxy-7-iodoquinoline-5-sulphonic acid) are described. The analytical factor (19·82) was established by gravimetric and thermogravimetric analyses and corresponds to calcium loretinate trihydrate. The precipitation is quantitative in the pH range 3·7 to 5·9, and on the semi-micro scale 5 mg to 50 mg may be determined with a mean error of 0·35%. The microgravimetric method reduces the minimum quantity of Ca determined to 0·2 mg with a mean error of 0·45%. By dissolving the calcium loretinate in an excess of ferric solution, 20 μg of Ca was determined spectrophotometrically with a mean error of 0·35%.　　　　M. F. C. LADD

827. Rapid determination of the essential constituents of a "fat" lime (CaO, CaCO$_3$). P. Remy-Genneté and B. Audouze (Lab. de Chim. Ind., Fac. des Sci., Clermont-Ferrand, France). *Bull. Soc. Chim. France*, 1956, **6**, 957.—In a fresh lime, CaO is determined by thermogravimetry (3 to 10-g sample) (*cf. Anal. Abstr.*, 1957, **4**, 398) and $CaCO_3$ by the Schroedter method; 1 hr. is required for the determination. In a partially hydrated lime, CaO and $CaCO_3$ are determined as before and $Ca(OH)_2$ is determined by loss of weight on ignition above 910°; 2 hr. is required. In a magnesian lime where Mg is determined titrimetrically, 4 hr. is required.
　　　　E. J. H. BIRCH

828. The rapid separation and determination of radio-strontium and radio-caesium in fission-product mixtures. J. C. Dalton and G. A. Welch (Atomic Energy Authority, Windscale, Cumberland, England). *Anal. Chim. Acta*, 1956, **15** (4), 317–319 (in English).—A procedure is described for the determination of ^{89}Sr and ^{137}Cs in the eluate after the removal of all other fission products from soln. by pptn. on an anion-exchange column in the hydroxide form (*cf. Nature*, 1956, **177**, 183). The Sr is first pptd. quant. as $SrCO_3$ by addition of solid CO_2 to the eluate and, after dissolution of the $SrCO_3$ in acid, is re-pptd. as oxalate. The Cs in the eluate can then be pptd. as $CsClO_4$. Both ppt. are weighed and counted immediately. For the 1·5-MeV β-rays of ^{89}Sr, a total absorber-thickness of 130 to 140 mg per sq. cm is used, and an additional correction is made for the growth of ^{90}Y (half-life = 61 hr.) by recounting the sample a few days later. The γ-rays from ^{137m}Ba ($\equiv$ only 82·5% of the total ^{137}Cs) are counted on a standard scintillation apparatus fitted with a lead - aluminium absorber (2 g per sq. cm). Addition of 10 mg of cerous nitrate (as carrier) to the soln. before passing it through the resin column prevents elution of radio-cerium that would otherwise cause a variable error in the results. About 8 determinations of each element can be made within 2 days, with an error of $\simeq 10\%$ for Sr and 8% for Cs.　　　　W. J. BAKER

829. Micro-analytical determination of barium sulphate. H. Ballczo and G. Doppler (Chem. Lab. Univ. Vienna, Austria). *Mikrochim. Acta*, 1956,

(4–6), 734–740.—Small quantities of $BaSO_4$ are decomposed by heating with $NaPO_3$ at 1000°. The $BaNaPO_4$ so formed is dissolved and the Ba^{2+} are complexed with EDTA, the excess of which is back-titrated with standard $MgCl_2$ solution; the indicator used is prepared by adding Tropaeolin OO to Eriochrome black T. This method allows 60 μg of Ba to be determined with a relative error of $\pm 3\%$.　　　　M. F. C. LADD

830. New ultra-micro methods for the direct determination of zinc on paper. A. Lacourt and P. Heyndryckx (Mikrochem. Dept., Univ. Brussels, Belgium). *Mikrochim. Acta*, 1956, (11), 1685–1704. —Titrimetric and spectrophotometric methods for the determination of ultra-micro quantities of Zn on paper before or after chromatography are compared. The reproducibility is improved by chromatographic development. The lower limit of sensitivity by titration with EDTA is 5 μg of Zn. The sensitivity is improved to 0·1 μg by a spectrophotometric procedure which involves comparison of the extinction at 520 mμ of the colour of the dithizone complex with that of a "paper blank." The precision is better than 0·3%. Eight determinations, including the paper chromatography, can be completed in 2 hr.　　　　A. R. ROGERS

831. Polarographic half-wave potentials. VI. Half-wave potentials of zinc. K. Micka (Polarogr. Inst., Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 203–211.—Half-wave potentials of Zn^{2+} in solutions of $KClO_3$ and $NaClO_3$, in relation to concn. of Zn, concn. of $ClO_3{}^-$, velocity of mercury flow and the drop-time, have been exactly measured. Various important values are given and theoretical conclusions derived.　　　　J. ZÝKA

832. Influence of pH on the extraction of zinc, cobalt, nickel and copper from tartrate solutions using dithizone. D. J. Eve and A. Strasheim (Nat. Phys. Lab., Council for Sci. and Ind. Res., Pretoria, S. Africa). *J. S. Afr. Chem. Inst.*, 1956, **9** (1), 5–11. —The effect of pH on the extraction of Zn, Co, Ni and Cu from aq. tartrate solutions with solutions of dithizone in chloroform was investigated. The relationship derived by Kolthoff and Sandell (*J. Amer. Chem. Soc.*, 1941, **63**, 1906) was found to be applicable to Ni and Co up to pH $\not> 8$ and equilibrium constants for the extraction of Ni, Co and Zn dithizonates are calculated to be, respectively, 1.06×10^{-8}, 3.48×10^{-7}, and 5.4×10^{-5}.
　　　　S.C.I. ABSTR.

833. Amperometric and potentiometric titrations of cadmium with ethylenediaminetetra-acetate using dropping-mercury electrode as indicator electrode. Nobuyuki Tanaka, I. T. Oiwa and Mutsuo Kodama (Dept. of Chemistry, Fac. Sci., Tohoku Univ., Sendai, Japan). *Anal. Chem.*, 1956, **28** (10), 1555–1559.—The current - voltage curves of Cd in an acetate buffer (pH 4·2) in the absence and presence of EDTA (disodium salt) in various proportions are given. Adams's work (*Anal. Abstr.*, 1955, **2**, 1436) was confirmed for the ratio of Cd to EDTA of 1:1, showing a small reducing current of free Cd^{2+}. The limiting currents of the reduction of free Cd^{2+} were markedly suppressed by the presence of gelatin (0·001%), which also affected the reduction wave of the Cd - EDTA complex. It is suggested that the wave corresponding to the reduction of free Cd^{2+} is due to the complex dissociating on the mercury surface and forming free Cd^{2+} in the course of electrolysis, and hence kinetic currents. In the presence

of gelatin there is good agreement between experimental end-points and the equivalence points. Potentiometric titrations were made of various concn. of Cd at const. current in an acetate buffer (pH 4·2), the ionic strength being made 0·1 with KNO_3. Potential jumps occurred before the equivalence points in all titrations, as expected. The deviation from the stoicheiometric end-point increased with decrease in Cd concn., and with increase in applied current. The amount of titrant required for the indicator electrode to obtain a value of − 0·700 V vs. the S.C.E. was taken as the experimental end-point. The theoretical deviation at this point can be calculated and may be applied to the practical titration to determine the initial concn. of substance. If the concn. is calculated by means of this relationship, potentiometric titrations can be applied to solutions of fairly low concn. ($M \times 10^{-3}$).
D. G. LLOYD

834. The determination of mercury in ores and concentrates. J. Michal, J. Jankovský and E. Pavlíková (Inst. für Erzforschung, Prague). *Z. anal. Chem.*, 1956, **153** (2), 83–88.—The method of Eschka for the determination of Hg in ores and slurries has been modified to permit compleximetric titration. *Procedure*—Mix the sample (containing 0·01 to 20% of Hg) with half its weight of iron powder and cover with a layer of ZnO. Heat in a porcelain crucible covered with a water-cooled gold lid, slowly for 10 min., then at a dull heat for 20 min. Allow to cool slowly, immerse the lid in HNO_3 (1:4) (30 ml) in a 250-ml beaker, bring to the boil and remove the lid, washing it well. Add 30% H_2O_2 (a few drops), boil, cool, add an excess of 0·05 M EDTA (disodium salt), neutralise to methyl red, add buffer of pH 10 (3 to 5 ml), dilute to 100 or 200 ml and titrate with 0·05 M $ZnCl_2$, with Eriochrome black T as indicator.
A. R. ROGERS

835. Spectrographic determination of mercury co-precipitated with tellurium; factors affecting conditions of excitation. H. Triché and M. Cros. *Compt. Rend.*, 1956, **242** (21), 2567–2569.—From 5 to 500 μg of Hg in 10 ml of soln. can be determined by quant. co-pptn. with Te in the presence of SO_2 or hypophosphorous acid, followed by spectrographic measurement of the intensity of the Hg line, 2536·5 A, when the compound TeHg is volatilised in the electric arc. A spark-excited intermittent arc between carbon electrodes is used, with a filter to diminish the intensity of the Te line (2530·7 A). Conditions influencing excitation are discussed. Fractional distillation of Te and Hg must be prevented (add a known amount of powdered C to the sample soln.) because it affects both element concn. and excitation conditions. The error is ≃7%.
W. J. BAKER

836. A new microchemical reaction for the detection and differentiation of mercury, iridium, indium, platinum and gold. B. Berisso (Inst. Invest. Microquím. Univ. Litoral, Rosario, Argentina). *Mikrochim. Acta*, 1956, (4–6), 812–821.—The characteristic micro crystal forms yielded by the reaction between 5% aq. methylene blue and Hg, Ir or In are directly distinguishable from one another under the microscope. Induced reactions may be used for Pt. With Pt and Au, CrO_4^{2-}, $Cr_2O_7^{2-}$ and ClO_3^{-} interfere. The limits of identification and concentration for Hg, Ir, In, Pt and Au are, respectively, 0·05 μg, 10^{-5}; 0·05 μg, 10^{-5}; 0·05 μg, 10^{-4}; 0·05 μg, 10^{-5}; 1 μg, 5×10^{-4}.
M. F. C. LADD

837. The polarographic determination of boron. D. T. Lewis (Atomic Weapons Res. Estab., Aldermaston, Berks., England). *Analyst*, 1956, **81**, 531–536.—Neither orthoboric acid nor any of the complexes formed with polyhydroxy compounds (e.g., mannitol) yields a polarogram with the dropping-mercury electrode. In the presence of mannitol, however, addition of Na_2SO_3 soln. causes the immediate formation of two distinct waves. It is established that these waves are due to liberation of free H_2SO_3 by the action of the relatively strong polyhydroxyboric acid. Qual. and quant. applications of this reaction are discussed.
A. O. JONES

838. Isotopic analysis of gaseous boron hydrides by neutron absorption. R. P. Hamlen and W. S. Koski (The Johns Hopkins Univ., Baltimore 18, Md., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1631–1632.—An isotopic analysis of diborane and pentaborane containing various amounts of ^{10}B, with a neutron-absorption technique, is described and should also be applicable to other gaseous boron hydrides. The accuracy and reproducibility of the results agree to within 1% with those obtained by mass spectroscopy.
K. A. PROCTOR

839. Applications of sensitivity diagrams. II. Semi-quantitative determination of impurities in borax. F. Burriel-Martí, J. Ramírez-Muñoz and R. Escobar-Godoy (Univ. Madrid). *An. Real Soc. Esp. Fis. Quim., B*, 1956, **52** (6), 417–434.—The procedure involves constructing sensitivity diagrams for a qualitative test for the impurity to be determined. These diagrams are then converted into dilution graphs from which the concn. of impurity may be derived, following successive dilution of a solution of the test substance down to the limit of sensitivity of the test. The application of the method to the semi-quantitative determination of Ca^{2+}, K^+, Mg^{2+} and Cl^- in borax, using various reagents, is described.
III. Semi-quantitative determination of sulphate in primary substances. F. Burriel-Martí, J. Ramírez-Muñoz and M. L. Rexach-M. de Lizarduy. *Ibid.*, 1956, **52** (6), 435–446.—The method is further applied to the determination of SO_4^{2-} in borax, KCl, KIO_3 and $KBrO_3$. The last two salts need to be converted into KCl by evaporation with conc. HCl before carrying out the sensitivity tests (with $BaCl_2$ soln.).
L. A. O'NEILL

840. Determination of aluminium with 8-hydroxyquinoline in the presence of iron, calcium and phosphoric acid. K. Gassner (Anorg. Forschungslab., Chem. Werke Albert, Wiesbaden-Biebrich/Rhein, Germany). *Z. anal. Chem.*, 1956, **152** (6), 417–419.—A simple and rapid method is described for the determination of Al in the presence of Fe, Ca and phosphate with an error of <1%. *Procedure*—Neutralise the soln. of the sample (containing 5 to 100 mg of Al_2O_3) with aq. NH_3 to a pH of 2 to 4. Stir in ascorbic acid (a few mg), a soln. of citric acid (4 g) in aq. NH_3 (10 ml), and NaCN (a few mg), in order to mask any Fe and Ca. If Mg is present, stir with ammonium phosphate (1 g) for about 3 hr. and remove the magnesium ammonium phosphate by filtering. Heat to 90° and add a slight excess of a 2% soln. of 8-hydroxyquinoline in 2 N acetic acid, dropwise, with stirring. Neutralise to the full red colour of phenolphthalein, allow to digest for 20 min., filter, wash the ppt. with hot, then cold, H_2O, dry for 1 hr. at 120° and weigh.
A. R. ROGERS

841. Determination of aluminium in chromic - phosphoric acid solutions. Determination of aluminium in aluminium corrosion products. C. Groot, R. M. Peekema and V. H. Troutner (Coatings and Corrosion Unit, Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1571–1576.—A method in which ammonium aurintricarboxylate (aluminon) is used is described for the estimation of Al in aluminium corrosion products after stripping with 2% chromic - 5% phosphoric acid. Interference from CrO_4^{2-} is removed either by pptn. of Al as phosphate or by the use of Dowex 1-X8 ion-exchange resin in chloride form. Interference by PO_4^{3-} is reduced if the concn. of Al is not less than 170 p.p.m., and by Fe^{3+} by reduction with thioglycollic acid. The extinction is measured at 530 mμ after 30 $\pm$ 0·5 min. at pH 4·4 to 4·75. Beer's law does not hold, but the calibration curve is reproducible. The accuracy is within $\pm3\%$ for samples containing 0·1 to 25·0 mg of Al. 　　　　　　　　　　　　D. G. LLOYD

842. Distinction of aluminium alloys according to the type of radiation given in electrolytes. V. A. Krasnokut-skiǐ. *Zavod. Lab.*, 1956, **22** (8), 960–961.—By connecting aluminium-alloy electrodes immersed in certain electrolytes (5 to 10% Na_2HPO_4 or $NaHCO_3$ solutions are recommended) to the lighting circuit and observing them in the dark, characteristic radiation resembling luminescence is observed. The colours (blue, yellow, etc.) can be compared with those given by alloys of known composition. With a.c., light is given by both electrodes, but with d.c. only by the anode. The method can be used under field conditions for sorting aluminium alloys. 　　　　　　　G. S. SMITH

843. Spectrophotometric determination of aluminium in ferrous and non-ferrous alloys. R. J. Hynek and L. J. Wrangell (Res. Lab., Allis-Chalmers Mfg. Co., Milwaukee, Wis., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1520–1527.—Many elements interfering in the estimation of Al may be removed as complexes with 8-hydroxyquinaldine (**I**), which does not react with Al. A procedure is given for acid-soluble and -insoluble samples containing up to 10% of Al. It consists in (*i*) separation of large amounts of base metals and other significant interferences from Al by mercury cathode electrolysis; (*ii*) use of **I** at pH 9·2 to remove residual traces of interfering elements; and (*iii*) spectrophotometric determination of Al as the 8-hydroxyquinolinate in $CHCl_3$ at 389 mμ. For amounts of Al $<0·1\%$, H_2O_2 is used to remove interference by Ti, Nb, Th, U and Ce. For amounts of Al in the range 0·1 to 10·0%, a modification of the method is used, which omits the mercury cathode electrolysis.

　　　　　　　　　　　　D. G. LLOYD

844. Reproducibility in the spectrographic analysis of aluminium alloys. F. H. Smith (ALAR, Ltd., 3 Albemarle St., London). *Metallurgia, Manchr*, 1956, **53**, 237–240.—The results are reported of a series of tests carried out to ascertain the order of reproducibility attainable in the routine analysis of aluminium alloys by means of a medium quartz spectrograph with photographic plates and a photometer. A comparison is also made with results obtained with a direct-reading Quantometer. The results confirm that the spectrograph is capable of giving routine results with a reproducibility comparable with that of chemical methods for most aluminium alloys, and with greater precision for contents of elements lower than normally found in aluminium alloys. 　　　　　S.C.I. ABSTR.

845. Determination of the ignition loss of hydrated alumina. H. Weile (Kali-Chemie A.-G., Hanover, Germany). *Anal. Chim. Acta*, 1956, **15** (4), 326–329. —In the determination of Al_2O_3 in samples of "pure" $Al(OH)_3$ and other hydrated aluminas, ignition at a temp. between 1200° and 1350° will give inaccurate results unless elaborate precautions are taken to prevent absorption of H_2O by the hygroscopic product. Constant wt. is attained only if the ignition is effected at 1500° or above.

　　　　　　　　　　　　W. J. BAKER

846. The separation of micro amounts of indium from zinc by liquid - liquid extraction. L. Kosta and J. Hoste (Univ. Ghent, Belgium). *Mikrochim. Acta*, 1956, (4–6), 790–796 (in English).—By using the radioisotopes ^{114}In and ^{65}Zn, it has been shown that whereas $ZnBr_2$ and $InBr_3$, both in aqueous HBr solution, are extracted by diethyl ether, when *iso*propyl ether is used the extractability of $ZnBr_2$, in the range 0·5 to 6 M HBr, is negligible. The use of *iso*propyl ether to separate Zn and In in the analysis of zinc blendes is suggested.

　　　　　　　　　　　　M. F. C. LADD

847. Determination of micro amounts of indium in zinc and in gallium by radioactivation. J. Hoste and H. van den Berghe (Univ. Ghent, Belgium). *Mikrochim. Acta*, 1956, (4–6), 797–803 (in English). —Activation of 10 g of Zn for 5 hr., with a 1-g Ra–Be thermal-neutron source, gave 17·4 counts per sec. per 10 g of Zn in a well-type sodium iodide scintillation counter due to the 13·8-hr. ^{69}Zn isotope. Activation of In under similar conditions gave 28·9 counts per sec. per 10 mg of In due to the 54-min. ^{116}In isotope. Thus the direct determination of 0·004 to 0·16% of In in a 10-g sample of Zn was carried out with a mean error of 2·3%. With somewhat less accuracy In may be determined in gallium after liquid - liquid extraction with diethyl ether. (*Cf.* Kosta and Hoste, *Anal. Abstr.*, 1957, **4**, 846.) 　　　M. F. C. LADD

848. Spectrophotometric determination of rare-earth mixtures. C. V. Banks and D. W. Klingman (Iowa State Coll., Ames, U.S.A.). *Anal. Chim. Acta*, 1956, **15** (4), 356–363 (in English).—The quant. analysis of mixtures of rare earths (as perchlorates) by using the characteristic mol. absorption spectra of the corresponding elements is discussed in relation to procedures and instrumentation. The mol. absorptions of Nd, Pr, Sm, Eu, Gd, Dy, Ho, Er, Tm and Yb have been re-determined at the appropriate wavelength in a Beckman DU spectrophotometer (with and without photomultiplier), and also in a Carey model 14 recording spectrophotometer (which ensures complete resolution of the rare-earth bands). The Beckman instrument cannot be safely used for determining Gd in a soln. containing Ce, or at wavelengths < 400 mμ, unless fitted with a photomultiplier to cover the range from 250 to 600 mμ. A general procedure for the routine analysis of a wide variety of rare-earth mixtures is outlined; the error is from 0·2 to 1%.

　　　　　　　　　　　　W. J. BAKER

849. A preliminary report on the determination of submicrogram quantities of individual rare-earths by radioactivation using ion-exchange separation. F. W. Cornish (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 1224, 1956, 44 pp.—The method is based on the general principles of radio-activation analysis, the separation of individual rare-earths being carried out by elution from a heated 100-cm column of Zeo-Karb 225. The method

is more sensitive than any other and can be used for (a) analysis of traces of rare earths in samples of limited wt. and at very low levels, (b) analysis of traces of rare earths in otherwise pure rare earths and (c) checking spectrographic results. Where duplicate analyses had been performed, agreement was usually reasonable and the results on a sample of graphite were in good agreement with spectrographic figures. Although the ultimate sensitivity had not been approached, some results were obtained which were below the limit of spectrographic detection, even although a sample of only one-tenth the wt. of that for the latter method was needed. The greatest difficulty lies in the separation of the rare earths by ion exchange and this can be very time-consuming, four days' work being needed to separate all the rare earths. The separation must also be performed simultaneously on a standard mixture. Because of this time factor some of the shorter lived isotopes had almost completely decayed before counting could be started.

K. A. Proctor

850. Separation of cerium from lanthanum by oxinesulphonic acid. Tiao-Hsii Chang (Nat. Taiwan Univ., Taipeh, Formosa). *J. Chinese Chem. Soc., II,* 1955, **2**, 63–70 (in English).—Tervalent rare-earth metals and oxinesulphonic acid (**I**) gave complex salts that were easily sol. in an excess of ammoniacal soln., of which that of Ce was oxidisable even by air (*cf.* Nakatsuka and Chang, *Acta Chim. Taiwanica,* 1948, **1**, 37). **I** (3 g) was neutralised with excess of aq. NH_3, diluted to 75 ml, and mixed with a soln. containing cerium nitrate (0·2241 g of CeO_2) and lanthanum nitrate. The mixture was oxidised with an O - NH_3 mixture at 55° for 3 hr.; Na_2CO_3 (10 g) was then added, the mixture was heated, and the carbonate ppt. was filtered off. The filtrate contained Ce (0·2201 g of CeO_2), but no La was found. An unsatisfactory result was obtained when O was replaced by air in the above procedure. An attempt to oxidise the remaining Ce complex (not oxidised by air) with H_2O_2 failed. The reactions involved are discussed.

Chem. Abstr.

851. The extraction of metal complexes. XXIV. The extraction of lanthanum, thorium and uranium (VI) with some phenyl[aryl]carboxylic acids. B. Hök-Bernström (Försvarets Forskningsanstalt, Sundbyberg, Sweden). *Svensk Kem. Tidskr.,* 1956, **68**, 34–39 (in English).—The separation of small amounts of La^{3+}, Th^{4+} and UO_2^{2+} by extraction with, *e.g.*, salicylic acid, cinnamic acid or 3:5-dinitrobenzoic acid, has been studied in the solvent systems *iso*butyl methyl ketone - H_2O and $CHCl_3$ - H_2O. The aq. phase is kept at constant ionic strength (0·1 M) by the addition of $NaClO_4$. Curves show the degree of separation of the three types of ion at 25° in terms of ligand-ion concn. A complete separation of Th^{4+} from La^{3+} is always possible, but very close control of extraction conditions is necessary for the effective separation of Th^{4+} from U^{VI}. *iso*Butyl methyl ketone is an excellent solvent for these organometallic complexes, but $CHCl_3$ is a very poor one. The three arylcarboxylic acids used form extractable complexes with Pu^{4+}.

W. J. Baker

852. Determination of lanthanum, iron and magnesium in uranium by flame spectrophotometry. J. F. Possidoni de Albinati. *An. Asoc. Quim. Argentina,* 1955, **43**, 106–120.—Impurities in U, such as La, Fe and Mg, can be separated from an aq. HNO_3 soln. of $UO_2(NO_3)_2$ by ether extraction,

and then determined by flame spectrophotometry. A 10-g sample of U is converted to the nitrate and the aq. soln. evaporated almost to dryness. The residue is placed in a separating funnel and dissolved in 4 M HNO_3. The soln. is extracted with 20 × 5-ml portions of peroxide-free ether, and after each extraction 2 ml of HNO_3 is added to the soln. to maintain the acidity. The extractions are continued until the ether phase is colourless. The aq. phase, which contains the La, Fe and Mg impurities, is heated gently to remove dissolved ether and then evaporated almost to dryness. If Fe is present it must be separated. The residue is therefore treated with 0·5 ml of H_2O and again evaporated. The residue is dissolved in 5 to 7 ml of 6 N HCl, and the $FeCl_3$ is removed by ether extraction in a separating funnel. The aq. phase containing La and Mg is evaporated almost to dryness, the residue dissolved in H_2O and made up to 25 ml. The ether phase containing the Fe is evaporated, the residue is dissolved in H_2O, and the vol. made up to 10 ml. A Beckman DU spectrophotometer, equipped with a flame attachment with either a H - O burner or an O - C_2H_2 burner is used to determine the quantities of La, Fe and Mg; La is determined at 440 mμ, Mg at 371 mμ, and Fe at 372 mμ. Calibration curves were prepared for each element and the effect of interfering ions was determined.

Chem. Abstr.

853. A simple and sensitive method for estimating radio-carbon (^{14}C). L. Sverak (Chem. Lab., Univ., Vienna). *Mikrochim. Acta,* 1956, (4–6), 1068–1074.—The Geiger counter tubes are filled with pure CO_2 and operated with an external quenching circuit. The yield is 80 to 90%. An anticoincidence device was built to lower the blank value by a factor of 10. Present methods of measuring ^{14}C are critically reviewed.

M. F. C. Ladd

854. Determination of carbon monoxide in small gas volumes. H. Linderholm and T. Sjöstrand (Dept. of Clin. Physiol., Karolinska Sjukhuset, Stockholm, Sweden). *Acta Physiol. Scand.,* 1956, **37** (2–3), 240–250 (in English).—A method for the determination of CO in small vol. of gas with hopcalite meters is described. The applicability of these meters has been greatly increased by the use of electrical temp. measurement and by the use of a recorder. About 0·001 ml to 1 ml of pure CO can be measured in gas vol. varying from 0·02 to 500 ml, irrespective of the CO concn. The coefficient of variation ranges from 0·5 to 3%, depending on the method of injecting the gas and the vol. used. Calibration is carried out by comparison with 100% CO. The method is especially suitable for the analysis of small amounts of CO extracted from blood samples.

K. A. Proctor

855. The accuracy and precision of "rapid methods" of silicate analysis. E. L. P. Mercy (Dept. of Geol., Imperial Coll. of Sci. and Technol., London). *Geochim. Cosmochim. Acta,* 1956, **9** (4), 161–173 (in English).—The "rapid methods" devised by Shapiro and Brannock (*U.S. Geol. Survey Circ.,* No. 165, 1952) for silicate analysis are discussed. The major modification proposed is that, in the determination of Fe, the yellow ferric chloride complex should be replaced by the ferrous iron complex with thioglycollic acid (Milner and Groom, *Metallurgia,* 1951, **44**, 271). It is also suggested that the titration of Ca should be avoided by using the photometric end-point determination devised by Chalmers (*Analyst,* 1954, **79**, 519). The accuracy

and precision of the methods are discussed and shown to compare very favourably both with conventional chemical and with spectrographic procedures. They are ideally suited for serial analysis of a rock complex and will give adequate comparative data of known precision on single analyses of individual rock samples, although it is advisable in such cases to repeat about 10% of the analyses. For odd analyses of silicate rocks, determinations should be at least in duplicate. By repeating an analysis four or more times great precision may be attained which will be at least as good as, if not better than, that attained by conventional chemical methods.　　　　　　　D. F. PHILLIPS

856. Titration of bivalent tin salts with hydrogen peroxide. J. Vulterin and J. Zýka (Inst. Anal. Chem., Karlova Univ., Prague, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 311–312.—A $0 \cdot 1 \, M$ solution of H_2O_2, when kept in darkness, does not change its titre during 8 hr. and can be used for the volumetric determination of Sn^{2+} ($Sn^{2+} + H_2O_2 + 2H^+ \rightarrow Sn^{4+} + 2H_2O$). The titration of 5 to 50 mg of Sn^{2+} can be carried out in conc. HCl, in an atm. of N, by a potentiometric arrangement; Sb^{3+} do not interfere.　　　　　J. ZÝKA

857. The separation and determination of traces of lead in the presence of small amounts of bismuth. V. J. Moore (Chem. Lab., Roan Antelope Copper Mines Ltd., Luanshya, N. Rhodesia). *Analyst*, 1956, **81**, 553–554.—Bismuth interfered with the determination of Pb in three methods that were investigated, but a method involving co-pptn. of Bi with CuS in 2 N HCl leaving the Pb in the filtrate proved satisfactory. After extraction of the sample soln. with chloroformic dithizone soln. at pH 11, the chloroform layer was evaporated to dryness, organic matter was destroyed and the residue was dissolved in 25 ml of 2 N HCl containing $0 \cdot 2$ g of Cu. The soln. was treated with H_2S before and after boiling, paper pulp was added and the liquid was filtered, the pptd. sulphides being washed with 2 N HCl saturated with H_2S. This procedure effected complete separation, and recovery of Pb from the filtrate was satisfactory.　　　　A. O. JONES

858. Determination of lead and its compounds in the active masses of lead accumulators. N. A. Filippova and T. F. Dubrovskaya (Filial of the Sci. Res. Accumulator Inst., Podol'sk). *Zavod. Lab.*, 1956, **22** (8), 907–911.—The extraction of $PbSO_4$ with NaCl soln. from the negative masses of a lead accumulator is incomplete because the PbO present reduces the solubility of $PbSO_4$. Prior extraction of PbO with 5% acetic acid soln. is recommended. A method for determining PbO, $PbSO_4$ and Pb or PbO_2 on a single sample is described. *Procedure*—The sample (2 to $2 \cdot 5$ g of the active mass of the charged plate, or 1 g of that of the discharged plate) is stirred for 15 min. with 100 ml of 5% acetic acid soln. The residue is collected, washed first with 5% acetic acid soln. and then with 40 to 50% aq. ethanol to remove soluble Pb, and retained for the determination of $PbSO_4$, Pb and PbO_2. The filtrate is diluted to 500 ml, either with 5% acetic acid soln. for the determination of Pb in an aliquot by ammonium molybdate titration, or with water for the determination of Pb with EDTA, in which case an aliquot (10 to 15 ml) diluted with 50 to 75 ml of water is mixed with 15 ml of a buffer soln. (prepared by neutralising 100 ml of 10% tartaric acid soln. to methyl red indicator with aq. NH_3, and adding 100 ml of a soln. containing $5 \cdot 4$ g of NH_4Cl

and 35 ml of 25% aq. NH_3) and Eriochrome black T indicator, and titrated with $0 \cdot 03 \, N$ EDTA. The filter containing the insol. matter is placed in the original flask and shaken energetically for 1 hr. with 100 ml of 20% NaCl soln. The insol. matter containing Pb or PbO_2 is collected and reserved. The filtrate is diluted to 250 ml and the lead content in an aliquot is determined by titration with molybdate or EDTA. The metallic Pb or PbO_2 is dissolved in 25 ml of dil. HNO_3 $(1 + 4)$ and, if necessary, a few drops of 30% H_2O_2, and the soln., diluted to 120 to 150 ml, is filtered. The filtrate is diluted to 250 ml with water in a calibrated flask, and an aliquot is titrated with molybdate or EDTA.
　　　　　　　G. S. SMITH

859. Determination of lead in paints at the one per cent. level. M. W. Westgate, R. B. Shurts and E. F. Adams (Nat. Paint Varn. Lacq. Ass. Inc., Washington, U.S.A.). *Circ. Nat. Paint Varn. Ass.*, 1956, No. 773, 8–17.—Results of co-operative tests on the determination of Pb in paints tinted with known amounts of lead chromate (in liquid and dried film form) by a standard chromate method, involving wet-ashing with HNO_3 - H_2SO_4, isolation of Pb as $PbSO_4$ and pptn. as $PbCrO_4$, are reported. An improved procedure involving wet-ashing with Caro's acid has been developed. After extraction of the $PbSO_4$ so formed with aq. ammonium acetate, a preliminary volumetric test on an aliquot of the solution to ascertain whether the content of Pb is likely to exceed 1% has been found advantageous. A known amount of standard ammonium molybdate solution is added with tannic acid as indicator; a yellow - brown colour shows that the end-point has been exceeded and that the Pb is <1%. If >1% of Pb is indicated, the amount is accurately determined on the remainder of the solution by pptn. as $PbCrO_4$.　　　　L. A. O'NEILL

860. Differential absorptiometry [of titanium]. W. T. L. Neal (Nat. Phys. Lab., Teddington, England). *Photoelect. Spectr. Gr. Bull.*, 1956, (9), 204–207.—The application of differential absorptiometry to the determination of Ti is described and the various errors involved are discussed. With this method for the determination of the pure metal the coefficient of variation is $0 \cdot 03\%$.　　K. A. PROCTOR

861. Spectrophotometric study of some analytically important complexes of titanium in solution. A. Okáč and L. Sommer (Univ. Brno, Czechoslovakia). *Anal. Chim. Acta*, 1956, **15** (4), 345–355.—Spectrophotometric analyses indicate the step-wise formation of complexes of Ti^{4+} with the following reagents in acid soln. at various pH values—chromotropic acid, catechol, gallic acid, sulphosalicylic acid (**I**), pyrogallolcarboxylic acid (**II**) and disodium catechol-3 : 5-disulphonate (Tiron). In dil. acid soln. a simple complex, TiR, is usually formed at pH <2, but in buffered soln. (pH 4 to 6) a relatively stable complex, TiR_3, is finally formed, except with **I** and **II**. There is also evidence of an intermediate complex, TiR_2, in some soln., stable only between pH of $\simeq 2$ and $2 \cdot 6$. The stability of these complexes is related directly to their absorption spectra and to the bond strength of the H atom in the ligand mol. The absorption max. of the complexes formed at pH $4 \cdot 5$ to $5 \cdot 5$ and also their mol. extinction coeff. are listed. The accuracy of some photometric procedures for the determination of Ti, *e.g.*, with gallic acid, chromotropic acid or tiron, is discussed.　　　　W. J. BAKER

862. Estimation of the acidities of titanium solutions. V. Damodaran (Nat. Chem. Lab., Poona, India). *J. Sci. Ind. Res., B, India*, 1956, **15** (5), 253–257.—Simple titrimetric procedures are described for the determination of "active" and "total" acids in mixed sulphate solutions of Ti, Fe and Al, and for an indirect determination of the Al in such soln. The "active" acid in a mixed sulphate solution of Ti, Fe^{II} and Al is determined after hydrolysis of the Ti by boiling and the addition of a neutral alkali oxalate to form a complex with Fe and Al. The "total" acid in the mixed sulphate solution is determined by the use of alkali tartrate to complex Ti, Fe and Al and to liberate their combined acids. Any Fe^{II} present is oxidised with neutral H_2O_2 before adding the tartrate. Large systematic deviations with soln. containing high concn. of Fe are reduced considerably by using an empirical correction factor. The addition of neutral KF, after titrating the soln. of the mixed tartrate complexes, breaks up the Al tartrate complex only, liberating an equiv. amount of titratable alkali. An excess of 5 to 10 times the stoicheiometric amount of complexing agents is generally used, though a much larger excess can be tolerated in some cases. I. JONES

863. Methods for analysing titanium sponge and intermediate products. P. R. Perry, R. W. Lewis and T. A. Sullivan (U.S. Bur. of Mines, Boulder City, Nev., U.S.A.). *U.S. Bur. Mines, Rep. Invest.*, 1955, **5168**, 45 pp.—This report gives tested methods for the analyses of $TiCl_4$, titanium sponge, and various intermediate materials and products. The methods are not claimed to be preferred or standard. For the analysis of titanium sponge, chemical procedures are given for the determination of Cl, N, Fe, C, Mg and Ca, Mn, Si, V, Mo, Cr, S, P, O and H, Ti, and Na (by flame photometry). Spectrochemical procedures are described for Fe, Mg, Mn, Si, Cr, V, and Al in titanium sponge. In the analysis of $TiCl_4$, chemical procedures are given for the determination of "free Cl," V, Fe, Si, S, P, H_2S and non-volatile residue. Spectrochemical procedures for V and Si in $TiCl_4$ are given, and also the methods for determining N and C in magnesium. CHEM. ABSTR.

864. The determination of oxygen in titanium. Vacuum fusion with a platinum bath. D. H. Wilkins and J. F. Fleischer (Gen. Electric Co., Schenectady, New York, U.S.A.). *Anal. Chim. Acta*, 1956, **15** (4), 334–336 (in English).—A procedure for the accurate determination of O in high-purity titanium by vacuum fusion at $\simeq 1850°$ with a platinum bath ($\simeq 25$ g) in a graphite crucible is described. In such a bath many more samples than usual can be analysed without interference from gettering and solidification, or the need of a blank for added Fe or Sn. The gas-collecting time is only 15 min., the blank correction being reduced to $\simeq 4$ p.p.m. on a 1-g sample. W. J. BAKER

865. Spectrographic determination of hydrogen in zirconium by the method of isotopic equilibrium. A. N. Zaĭdel' and K. I. Petrov. *Zavod. Lab.*, 1956, **22** (8), 923–926.—A known amount of the sample of Zr, *e.g.*, 1 g, is heated in deuterium at 800° for 15 min. and the isotopic composition of the gas is determined spectrographically. Contents of H in Zr of $1 \times 10^{-4} \%$ can be determined with a mean square error of $\pm 10\%$. The results are in good agreement with those obtained by vacuum extraction. G. S. SMITH

866. New reagents for determination of thorium. I. Hydroxy and methoxy derivatives of cinnamic acid. M. R. Verma, S. D. Paul and K. C. Agrawal (Nat. Phys. Lab. of India, New Delhi). *Z. anal. Chem.*, 1956, **152** (6), 427–433 (in English).—The 2-, 3- and 4-hydroxy, 4-methoxy, and 3:4-dimethoxy derivatives of cinnamic acid have been investigated for the separation and pptn. of Th alone or in the presence of tervalent rare earths. 4-Hydroxycinnamic acid gives a granular ppt. and is recommended. If separation from Ce^{IV} is required, the methoxy derivatives should be used since they simultaneously reduce Ce^{IV} to Ce^{III}. *Procedure*—Add an ethanolic soln. of the reagent (in fourfold, excess) to the boiling soln. of the sample, adjust to a pH of 3·8 to 4·0 by cautious addition of aq. NH_3, add KCl (0·5 g), boil for 5 min., allow to digest on a water bath for 90 min., filter through a Gooch crucible, wash the ppt. with a dilute soln. of the reagent, ignite and weigh as ThO_2. A. R. ROGERS

867. Analytical applications of the reaction of thorium with benzenephosphonic acid and its derivatives. R. J. Davis (Dept. of Chemistry, Iowa State Coll., Iowa, U.S.A.). *Iowa St. Coll. J. Sci.*, 1956, **30** (3), 345–346.—A new gravimetric method is described for the determination of Th. *Procedure* —To a boiling solution containing 0·04 to 0·3 g of Th and 150 ml of conc. HCl is added 50 ml of a 2·5% solution of benzenephosphonic acid. After boiling for 1 hr., the mixture is cooled, diluted to 500 ml with water, and the pH is adjusted to between 0·5 and 1·0 with aq. NH_3. After being set aside overnight, the ppt. is collected, washed six times with HCl (pH 0·5 to 1·0), dried to constant wt. at 140° to 180° and weighed; each gram of residue [$Th(C_6H_5PO_3)_2.3H_2O$] is equiv. to 0·38793 g of Th. The average error in 22 determinations was $+0·6$ part per thousand and the probable error $+3·6$ parts per thousand. Of 30 cations tested at molar concn. up to twice that of Th, serious interference occurred only with Ag^I, Tl^I, Ti^{IV}, Sb^{III}, Fe^{III}, Zr^{IV}, Sn^{IV} and U^{VI} ($>1\%$), and larger amounts of Ce^{IV}; Cl^-, ClO_4^-, NO_3^- and SO_4^{2-} did not interfere. Absorption spectra data of $C_6H_5PO_3^{2-}$, $C_6H_5PO_3H^-$, $C_6H_5PO_3H_2$ and $C_6H_5PO_3H_3^+$ are reported. S. C. JOLLY

868. Use of complexones in chemical analysis. XLVIII. Gravimetric determination of thorium. P. Schneider (Development Div., Tesla-Vršovice, Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1956, **21** (4), 1054–1056 (in English).— Thorium may be pptd. from soln. containing EDTA by aq. NH_3 in the presence of 5 to 10 ml of 30% H_2O_2. The peroxyhydrate ppt. can either be converted into the oxide, or dissolved in HCl and the Th determined compleximetrically. Although Th can be separated from U and many cations such as Fe and Al by a single operation, it cannot be separated from Ti and the rare earths. The method was applied to the determination of ThO_2 and Th in cathodes of thoriated tungsten. P. S. STROSS

869. Analytical aspects of some azo dyes derived from chromotropic acid. V. Gravimetric determination of thorium and zirconium with 1:8-dihydroxy-2-(4-sulphamylphenylazo)naphthalene-3:-6-disulphonic acid. S. K. Datta (Chem. Dept., Gov. Coll., Darjeeling, India). *Z. anal. Chem.*, 1956, **153** (2), 89–95 (in English).—Thorium and zirconium can be determined gravimetrically by pptn. of their complexes with 1:8-dihydroxy-2-(4-sulphamylphenylazo)naphthalene-3:6-disulphonic acid

(I) and ignition to the oxides. These metals cannot be separated from one another by this method but can be separated from Cu, Cd, Co, Ni, Mg, Ca, Sr, Ba, Au, U and the rare-earth metals, but not from Hg^I, Pb, Fe^{III}, Al or Cr. *Procedure*—Neutralise the soln. (containing > 5 mg of ThO_2 or ZrO_2) to Congo red, heat to 70° and add a 2% soln. of **I** in hot H_2O. Allow to digest on a water bath for 15 min., filter, wash the ppt. with hot H_2O, ignite and weigh as ThO_2 or ZrO_2.   A. R. ROGERS

870. Determination of nitrate with phenarsazinic acid. R. Pietsch (Inst. für anorg. und anal. Chem., Univ. Graz, Austria). *Mikrochim. Acta*, 1956, (11), 1672–1678.—Traces of nitrate can be determined absorptiometrically by means of the intense red colour given with phenarsazinic acid in alkaline soln. (*cf.* Pietsch, *Anal. Abstr.*, 1956, **3**, 3603). Few anions interfere; halides should be removed by pptn. with Ag_2SO_4. Interfering cations can readily be removed by use of an ion-exchange resin. *Procedure*—Mix the sample with a 0·3% soln. of phenarsazinic acid in 0·06% aq. NaOH (1 ml) and conc. H_2SO_4 (1 ml). Heat for 10 min. in a bath of boiling water, wash into 30% aq. NaOH (20 ml), dilute to 100 ml, mix, and measure the extinction against a reagent blank. Beer's law is obeyed in the range 10 to 70 μg of NO_3^-.   A. R. ROGERS

871. Rapid colorimetric method for nitrates. M. H. Swann and M. L. Adams (Paint and Chemical Lab., Aberdeen Proving Grounds, Md., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1630.—The brown-ring test for nitrates has been found to be applicable as a quantitative method of analysis for nitrates and also for some organic materials such as nitrocellulose in the presence of many anions. A solution of $FeSO_4.7H_2O$ in H_2SO_4 is added to the sample (an aliquot of an aq. soln. containing 0·5 to 2·5 mg of NO_3^-, dried at 105°, or of an acetone soln. dried at 60°) in an Erlenmeyer flask and shaken for a short period. The extinction at 525 mμ is measured against the reagent as blank. Both reagent and colour are stable for some hours. Nitrite and thiosulphate interfere. The method is not applicable to the analysis of lacquer coatings owing to interference by oils and some plasticisers, but has been applied to certain phosphate coating materials.   D. G. LLOYD

872. Rapid determination of nitrate nitrogen in the presence of ammonia and urea. R. M. Engelbrecht and F. A. McCoy (Lion Oil Div., Monsanto Chemical Co., El Dorado, Ark., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1619–1621.—Nitrate nitrogen may be determined in the presence of K^+, Na^+, SO_4^{2-}, PO_4^{3-}, NH_4^+ and urea by an oxidimetric method by using an excess of ferrous sulphate and back-titration with permanganate. Add 25 ml of N $FeSO_4.7H_2O$ (270 g of $FeSO_4.7H_2O$, 40 g of NaCl and 100 ml of conc. H_2SO_4 made up to 1 litre) to an aliquot of sample (25 ml containing $\not>$ 0·3 g of NO_3^-) in an Erlenmeyer flask. Immediately before the determination add conc. H_2SO_4 (20 ml) and heat for 3 min. until an orange colour appears. Titrate immediately with standard $KMnO_4$ (0·5 N), running a blank concurrently with the sample. In the presence of urea, add NaCl (5 g) to the sample and NaCl (5 g) and urea (1 g) to the blank. The method is well suited to determinations in mixed fertilisers and may be completed in 15 min. with an accuracy within $\pm 0.3\%$ for pure nitrate and within $\pm 1\%$ for nitrate in the presence of urea.
D. G. LLOYD

873. Microbiological determination of nitrate. G. B. Garner, J. S. Baumstark, M. E. Muhrer and W. H. Pfander (Univ. of Missouri, Columbia, Mo., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1589–1591.—The method is based on the reduction of NO_3^- to NO_2^- by using a nitrate reductase prepared from the rumen of a sheep; the nutritional requirements of the micro-organisms are met by trypticase. The NO_2^- are determined colorimetrically with N-1-naphthylethylenediamine - sulphanilic acid reagent at 550 mμ. The reproducibility is within ± 0.12 μg over the range 2 to 20 μg of KNO_3, and the method was successfully applied to silage, forage, hay, rumen fluid and urine samples.
G. P. COOK

874. Determination of water in fuming nitric acid by near-infra-red absorption. L. White, jun., and W. J. Barrett (Southern Res. Inst., Birmingham, Ala., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1538–1542.—For water contents up to at least 6% and nitrogen dioxide up to about 20% the near i.r. absorption of fuming HNO_3 at 1·423 μ is an almost specific measure of its water content. The method described for such determinations involves the use of a Beckman DU spectrophotometer modified by replacing the phototube housing and amplifier by an interchangeable unit comprising a 300 c/s chopper, a lead sulphide photoconductive cell and a tuned amplifier. Corrections are given for the effect of nitrogen dioxide, which is negligible unless both the water and nitrogen dioxide contents are low. The determination is not significantly affected by temp. within the normal range of laboratory temp. nor by the presence of dissolved nitrates. Determination on 30 samples containing up to 5% of water on each of four instruments showed variations among the instruments of $\not> 0.2\%$ water equivalent.
K. A. PROCTOR

875. Analytical determination of trace constituents in metal-finishing effluents. XI. Determination of nitrate - nitrite nitrogen. E. J. Serfass and R. F. Muraca (Lehigh Univ., Bethlehem, Pa., U.S.A.). *Plating*, 1956, **43**, 233–234.—In the procedure recommended, the sample (100 ml) is made alkaline, boiled down to $\simeq$ 20 ml (to remove NH_3) and washed into a Kjeldahl flask. The NO_3^- and NO_2^- are reduced (8 hr. at $\not< 20°$) by aluminium foil in alkaline soln., aq. Na_2S is added and the NH_3 is then removed by distillation, 50 to 60 ml of distillate being collected in 20 ml of 0·1 N HCl. The soln. is made up to 100 ml; a 10-ml aliquot is taken, 3 drops of 2% gum acacia soln. are added, followed by 1 ml of Nessler reagent. After thorough mixing and dilution to 100 ml with H_2O, a portion of the coloured soln. is transferred to a spectrophotometric cell and the extinction is measured at 400 mμ, with a blue filter. After deducting the blank value, the amount of N is read from a calibration curve. The sensitivity is 2 p.p.m. ($\equiv$ 200 μg of N in 100 ml of sample) and the limit of detection is 0·5 p.p.m. Organic compounds that react with NH_3 or generate NH_3 on reduction or hydrolysis in basic soln. should be absent. A shorter procedure is given for effluents of known composition (alkali and alkaline-earth metals, Zn, Al < 100 p.p.m., all other elements < 10 p.p.m., CN^-, SCN^- and sulphides < 1 p.p.m.).
W. J. BAKER

876. Qualitative detection of nitrite by means of paper chromatography. K. Täufel and R. Serzisko (Inst. für Ernährungsforsch., Potsdam-Rehbrücke, Germany). *Ernährungsforschung*, 1956, **1** (1), 149–151.—The detection of NO_2^- is based on the use

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of *n*-butanol - pyridine - 1·5 N aq. NH_3 (40:20:40) as ascending solvent, in which NO_2^- show an R_F value of 0·25. After development for 12 to 16 hr. the chromatogram is dried for 5 to 10 min. in a current of air at room temp., and then sprayed with a soln. containing 0·25 g of 1-naphthylamine in 99 ml of acetone plus 1 ml of 4 N H_2SO_4. The presence of NO_2^- ($\not< 5$ μg) is revealed by a red coloration. An example is given of the detection of NO_2^- in a filtered and clarified cold-water extract of minced pickled meat. P. S. ARUP

877. Indirect colorimetric determination of antimony. Tôru Nozaki (Faculty of Eng., Ehime Univ., Niihama, Ehime Prefecture). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (6), 960–962.—Quinquevalent Sb (< 1 mg) is quant. pptd. with *trans.*-dichlorotetra-amminocobalt chloride in 2 N HCl (Belcher *et al.*, *J. Chem. Soc.*, 1952, 4775). The ppt. is dissolved in HCl (1 + 1) and water, treated with Na_2S and evaporated with conc. HNO_3. The product is made up to a definite vol. and Co is determined colorimetrically with nitroso-R salt. No interference results from the presence of Tl^{3+} (< 10 times the weight of the Sb), Fe (< 5 times), As (< 10 times), Cu (< 11 times), Cd (< 5 times) or of Zn (< 13 times). K. SAITO

878. The determination of small amounts of bismuth in copper mattes and concentration products. V. J. Moore (Chem. Lab., Roan Antelope Copper Mines Ltd., Luanshya, N. Rhodesia). *Analyst*, 1956, **81**, 555–556.—In the electrolytic method described, the stage of wet-ashing of the filter-paper with CuS and Bi_2S_3 in the usual procedure is avoided. The sample is fumed with HNO_3 and H_2SO_4, with a special procedure if sulphides are present. The residue is boiled with dil. HNO_3 and, after removal of insol. matter, the filtrate is electrolysed until deposition is complete, $Cu(NO_3)_2$ soln. is added and electrolysis is continued to provide a protective sheath of Cu. The deposit is dissolved from the cathode with HNO_3, $Fe(NO_3)_3$ soln. and an excess of aq. NH_3 are added and the mixture is boiled. The liquid is filtered, the copper complex is washed away with a mixture of ammonium nitrate, ammonium carbonate and aq. NH_3. The ppt. is dissolved in hot dil. H_2SO_4, KI soln. is added, the liberated I is removed with a slight excess of SO_2 soln. The liquid is boiled, Se and Te (if present) are removed by filtration, and sodium hypophosphite is added to remove the colour of I, leaving the yellow colour of BiI_3. After dilution, the Bi is determined by means of a Spekker absorptiometer, with violet filters. Quoted results indicate satisfactory accuracy. A. O. JONES

879. Photocolorimetric determination of vanadium and titanium. E. F. Pen'kova, A. V. Gladkova and T. V. Novikova ("Élektrostal'" Works). *Zavod. Lab.*, 1956, **22** (8), 918.—Vanadium is determined in steel containing Mo, Mn, Cr and Ti by the reaction with H_2O_2. Titanium is determined by means of chromotropic acid. G. S. SMITH

880. The colorimetric determination of niobium with tiron. H. Flaschka and E. Lassner (Inst. f. anorg. Technol. u. anal. Chem. Tech. Hochschule, Graz, Austria). *Mikrochim. Acta*, 1956, (4–6), 778–783.—Both Nb^{III} and Nb^V form complexes with tiron, but the complex with Nb^{III} is not stable in air. The yellow complex of Nb^V is stable in acid (5 N HCl) and alkaline soln. and may be determined colorimetrically, preferably in acid solution.

Quinquevalent V interferes, but may be removed by reduction with ascorbic acid. The mean error in determinations of 2·5 to 8·5 mg of Nb in 50 ml of soln. is 0·1 mg. M. F. C. LADD

881. Separation of niobium and tantalum by paper chromatography. E. Bruninx, J. Eeckhout and J. Gillis. *Mikrochim. Acta*, 1956, (4–6), 689–699.—The micro-scale separation of complex oxalates of Nb and Ta on Whatman No. 1 paper has been studied with tracers of radioactive ^{95}Nb and ^{182}Ta. A very satisfactory eluent is ethyl methyl ketone - 10 N HCl (75:25), R_F values being 0·11 for Ta and 0·78 for Nb. The same eluent separates Ti and Nb from Fe; Ta, Nb and Fe; and also, with a cellulose column, Nb and Ta on a semi-micro scale. The purity of the Nb obtained was >99·9%. M. F. C. LADD

882. A method for the determination of traces of ozone. W. Deckert (Hygienische Inst. der Freien und Hansestadt, Hamburg, Germany). *Z. anal. Chem.*, 1956, **153** (3), 189–193.—A simple and rapid method is described for the detection and determination of traces of ozone in air, based on the reddening of Fe_2O_3 - KSCN paper (*cf. Anal. Abstr.*, 1956, **3**, 3206). The lower limit of detection is 10 μg of ozone in 10 litres of air; ozone concn. in the range 0·01 to 10 mg per litre can be determined with an accuracy of ±10% by measuring the area of paper reddened by the stream of sample gases. Nitric oxide does not interfere; H_2O_2 and acids also cause reddening, and must first be removed. A. R. ROGERS

883. Micro-determination of sulphur by the nuclear reaction ^{32}S (d, α) ^{30}P. P. Süe and P. Albert. *Compt. Rend.*, 1956, **242** (20), 2461–2462.—The determination of 10 p.p.m. or 1 μg of S can be effected, with an error of $\simeq \pm 15\%$, by deuteron irradiation of the sample ($\simeq 20$ mg) for 7 min., followed by a double pptn. of ^{30}P in aq. $Mg(NH_4)PO_4$ soln. and a count of the activity of the ppt. 10 min. after the irradiation. There is no interference by the simultaneous formation of ^{34}Cl; 1 μg of S $\equiv \simeq$ 340 pulses per min. of ^{30}P. W. J. BAKER

884. Iodimetric determination of total sulphur in pyrites. V. Šindýlek (Chem. Works, Přerov, Czechoslovakia). *Chem. Průmysl*, 1955, **5** (10), 433–435.—When burned with an excess of zinc filings in an atmosphere of CO_2, sulphur in pyrites is converted into sulphide, which can, after distillation, be determined iodimetrically. The detailed procedure is given and the interference from arsenic as well as the accuracy of the method compared with that of other common methods are discussed. J. ZÝKA

885. Thermometric determination of small amounts of sulphuric acid in chlorosulphonic acid. K. Kámen (Spolana, Neratovice, Czechoslovakia). *Chem. Průmysl*, 1955, **5** (5), 198–200.—When SO_3 is treated with gaseous HCl, an exothermic reaction takes place. The measurement of the increase in temperature was used for thermometric determination of small amounts of H_2SO_4 (even 0·1%) in chlorosulphonic acid. Apparatus and the detailed procedure are described. J. ZÝKA

886. Turbidimetric determination of sulphate in phosphoric acid and phosphates. K. Gassner and H. Friedel (Anorg. Forschungslab., Chem. Werke Albert, Wiesbaden-Biebrich/Rhein, Germany). *Z.*

anal. Chem., 1956, **152** (6), 420–424.—A rapid method is given for the turbidimetric determination of sulphate (0·01 to 2% of SO_3) in technical phosphoric acid and phosphates with an accuracy of $\pm 3\%$. Condensed phosphates must first be hydrolysed to the ortho form. *Procedure*—Adjust the soln. (containing 0·4 to 1·6 mg of SO_3) to a pH of 1 to 2, dilute to 40 ml, add 0·5% aq. gelatin (5 ml), mix well and slowly add a 2% soln. of $BaCl_2.2H_2O$ in 0·5% aq. gelatin (5 ml), with constant stirring. Prepare standard and blank soln. similarly. Compare the extinctions in 2-cm cells with the use of filter S53.   A. R. ROGERS

887. The rapid determination of sulphate in sodium chloride solution for electrolysis. Sinsaku Matui, Tadao Ueda and Sigeru Katumata (Asahi Denka Kogyo Co., Arakawa-ku, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (6), 591–592.—The volumetric methods for $SO_4{}^{2-}$ determination with EDTA (disodium salt) and with $BaCrO_4$ (Hinman's method) were examined and improvements are suggested. The indication of the end-point for the former method is markedly improved by the addition of methyl red and ethanol ($\simeq 5$ ml for 50 ml of the sample soln.). In the second method, on the neutralisation of the acid sample soln. containing an excess of $BaCrO_4$, a part of the pptd. $BaSO_4$ dissolves to produce Na_2SO_4 and $BaCrO_4$, and so gives low results. It is recommended that the $BaSO_4$ should be filtered off before the addition of aq. NH_3 soln.   K. SAITO

888. Conditions of formation and photocolorimetric determination of the tungsten - thiocyanate complex. D. N. Finkel'shteïn (Central Lab., Ural Geological Dept.). *Zavod. Lab.*, 1956, **22** (8), 911–915.—The optimum conditions for the photocolorimetric determination of W as the thiocyanate complex with reduction by $TiCl_3$ are studied. A violet colour-filter (300 to 440 mμ) practically eliminates the colour of Ti^{3+}, permits higher concn. of W to be determined and results in calibration curves that follow Beer's law. Because of the safe use of higher concn. of Ti^{3+} the colour of the soln. is rendered more stable. In H_2SO_4 soln. the max. colour intensity occurs at a fairly critical acidity of $4N$ and the colour is stable for 2 to 3 hr. In HCl soln. the max. colour is obtained at an acidity of $2N$, excess acidity has little effect, and the colour is stable for 4 to 5 hr. Traces of Fe or Mo in HCl soln. interfere unless the acidity is $\not< 3.1$ to $3.3N$. The instability const. of the tungsten complex is between 2.3×10^{-2} and 3.2×10^{-2}. The molar extinction coeff. at 380 mμ is between 5900 and 6500. In the presence of Mo a correction varying with the content of WO_3 can be made. Thus, the true content of WO_3 (mg in 50 ml) $=$ apparent content of WO_3 (mg in 50 ml) $- k \times$ content of Mo (mg in 50 ml), where k is 0·018 for 0·01 to 0·25 mg, 0·014 for 0·25 to 0·75 mg, 0·010 for 0·75 to 1 mg, and 0·005 for > 1 mg of WO_3 in 50 ml. Sexavalent Cr must first be reduced to Cr^{3+}, *e.g.*, by ethanol or H_2O_2 followed by $KMnO_4$. The colour is green, but a correction, which is independent of the content of WO_3, can be made. The coeff. is 0·005 for 0·5 to 10 mg of Cr_2O_3 and 0·01 for greater amounts in 50 ml of soln.   G. S. SMITH

889. The separation of tungsten and vanadium. B. Řehák and M. Malínek (Chem.-technol. Hochschule, Prague, Czechoslovakia). *Z. anal. Chem.*, 1956, **153** (3), 166–168.—Small amounts of W (5 to 50 mg) can be determined by pptn. as oxinate

in the presence of excess of V (10 to 1000 mg) by masking the V with EDTA; V^V must first be reduced to V^{IV}, *e.g.*, by heating with hydrazine sulphate. Small quantities (50 mg) of Al, Cu, Cr, Fe, Mn, Ni, Pb or Zn do not interfere. *Procedure*—Add a sevenfold excess of EDTA (tetrasodium salt) to the neutral sample soln. (100 ml), then aq. ammonium acetate (50%) (3 to 5 ml); heat, precipitate the W with a slight excess of 3% 8-hydroxyquinoline acetate soln., filter through a Jena G3 sintered-glass crucible, wash the ppt. with hot H_2O, dry at 120° to 140° and weigh as $WO_2(C_9H_6NO)_2$.
                    A. R. ROGERS

890. The polarographic determination of uranium. H. I. Shalgosky (U.K. Atomic Energy Authority, Woolwich Outstation, England). *Analyst*, 1956, **81**, 512–518.—The method of Legge (*Anal. Abstr.*, 1955, **2**, 624) for the polarographic determination of U in an acid tartrate medium has been examined and shown to give low results. Satisfactory results are obtained when $HClO_4$ is used in the preliminary chemical treatment and the necessary H_2SO_4 is added as dilute acid immediately before the polarographic determination. The sensitivity of the modified method is shown to be similar to that of other polarographic methods for the determination of U, and results with samples of known uranium content agreed with those obtained absorptiometrically. Factors affecting the sensitivity are discussed.   A. O. JONES

891. Determination of uranium as uranyl iodate. M. Venugopalan (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1956, **153** (3), 187–188 (in English). —Uranium can be determined by quant. pptn. as uranyl iodate; mineral acids and free Cl^- or NO_3^- must be absent. *Procedure*—Heat the sample soln. to 60°, add a known excess of saturated $NaIO_3$ soln. with stirring, cool, filter through a Gooch crucible and wash the ppt. with H_2O. Dry the ppt. at 110° and weigh as $UO_2(IO_3)_2$, or dissolve it in HCl and titrate with standard thiosulphate; or titrate the filtrate and washings.   A. R. ROGERS

892. The determination, in the presence of iron, of uranium in Gafsa (Tunisia) phosphate. R. Lumbroso, J. Petit and J. Spiteri. *Compt. Rend.*, 1956, **242**, 904–905.—In the procedure outlined, the Fe in the HCl soln. of the sample is first determined, after removal of most of the other metals except U. A series of standard soln. containing this amount of Fe and progressively increasing known amounts of U are then prepared, and the activity of each of these soln. is measured with a Geiger counter, a linear curve being constructed from the values obtained. If the activity of the sample soln. is determined, the U_3O_8 content ($\simeq 0.002$ to 0.005%) can be obtained from the standard curve.
                    W. J. BAKER

893. Micro-analytical studies of the oxyhalogen ions by chromatography and infra-red absorption. Y. Servigne (Lab. Recherches Micro-analytiques, 11 rue Pierre Curie, Paris). *Mikrochim. Acta*, 1956, (4–6), 750–759.—The lack of sensitive reactions for identifying anions as compared with cations is stressed. Partition chromatography was applied to the oxyhalogens and the R_F values were found; chlorates, bromates and iodates are separated with a mixture of *iso*propyl alcohol (75 ml) and water (25 ml) and the separation of chloride, chlorite and chlorate in admixture with hypochlorite can be effected. The reaction of the oxyhalogens with

thiocarbohydrazide was studied by chromatography on a kaolin column and with the aid of the microscope; characteristic crystal shapes are given. The infra-red absorption spectra of the oxyhalogens were obtained and it was found that chlorates could be detected in the presence of iodates and bromates. M. F. C. LADD

894. Spectrophotometric determination of fluoride. A comparative study and a suggestion for evaluating photometric data. B. Sen (Chem. Engng Dept., Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1956, **153** (3), 168–176 (in English).—Attempts to develop a method for the determination of F^-, in which the demasking principle was used, were unsuccessful. By measuring the fading of a coloured complex due to formation of a colourless fluoro complex, the iron - sulphosalicylic acid, iron - ti - ferron (tiron), Ti - chromotropic acid and Zr - alizarin systems have been studied as reagents for the determination of F^-. The accuracies of the methods were compared by examining the slopes of curves of differences in percentage transmission between the soln. of unit concn. and soln. of other concn. plotted against log concn. The iron - sulphosalicylic acid system is recommended; the coloured stock soln. is stable, and the results are accurate and reproducible. *Procedure*—Adjust the sample soln. (containing 0·8 to 4·0 p.p.m. of F^-) to a pH of 3·0 to 3·1, add a soln. of Fe^{3+} (80 p.p.m.) and sulphosalicylic acid (threefold excess) in buffer of pH 3·0 to 3·1 and measure the transmission at 500 mμ. The concn. of F^- is an inverse function of the extinction. A. R. ROGERS

895. Colorimetric determination of fluoride in water by heteropoly blue system. R. P. Curry and M. G. Mellon (Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1567–1570.—The fluoride content of water may be determined by distillation with H_2SO_4 (sp.gr. 1·84), with soft glass beads as a source of Si. A stream of nitrogen gas carries the SiF_4 into a $Na_3BO_3 - H_3BO_3$ buffer. The soluble silicate formed is converted into molybdosilicic acid and estimated colorimetrically after reduction by the heteropoly blue product. The standard deviation is 0·024 mg in the range 0·1 to 2·0 mg of F^-. Borate interferes if present in more than 20-fold excess. D. G. LLOYD

896. Determination of small amounts of fluorine in aqueous solutions. M. G. Lubyanskaya (Uzbek Sci. Research Inst. of Health and Physiotherapy). *Zavod. Lab.*, 1956, **22** (8), 921.—To determine 0·01 to 0·1 mg of F in water, the sample (40 ml) in a flat-bottomed cylinder (25 cm high × 2 cm wide) is mixed with 1 ml of 0·01% aq. Na alizarinsulphonate. By addition of 0·05 N acid or alkali the colour is made to match that of a soln. in a similar tube prepared from twice-distilled water. Then 2 ml of 0·1 N HCl is added and the soln. is titrated slowly with 0·001 M zirconium nitrate to a permanent pink colour. A calibration curve is constructed by titrating various amounts of a soln. of NaF (1 ml $\equiv$ 1 mg of F) diluted to 40 ml; its slope depends on the temp. and on the concn. of SO_4^{2-}. Any SO_4^{2-} in the sample must be first determined and a corresponding amount added to the standard NaF soln. G. S. SMITH

897. The qualitative and quantitative determination of chlorite. N. Konopik and E. Werner (I Chem. Lab., Univ. Vienna). *Öst. ChemZtg*, 1956, **57**, 49–50.—In order to eliminate errors in the polaro-

graphic determination of chlorite solutions, magnesium chloride is added. Practical details of the method are given. D. P. FELIX

898. Colorimetric determination of small amounts of manganese. Anon. Shirley Inst. Test Leaflet: No. Chem. 22, 1956, 6 pp.—Two methods are given, the second being more sensitive. Samples are first prepared by wet- or dry-ashing, followed by removal of interfering ions. In the first method, the Mn is oxidised with KIO_4 and the extinction of the resulting permanganate soln. is measured. The content of Mn is found by reference to a previously prepared calibration curve. A blank soln. is treated similarly. The second method, which is more complicated, depends on the measurement of the extinction of the brown complex formed by Mn with formaldoxime in alkaline soln.
A. M. SPRATT

899. Photometric determination of iron in phosphate and condensed phosphates with 2 : 2′-dipyridyl. K. Gassner (Anorg. Forschungslab., Chem. Werke Albert, Wiesbaden-Biebrich/Rhein, Germany). *Z. anal. Chem.*, 1956, **153** (2), 81–83.—Small amounts of Fe can be determined in the presence of a large excess of phosphate by reaction with 2 : 2′-dipyridyl (**I**) at an elevated temp.; the error is $\pm 1·5\%$. The lower limit of detection is 0·1 p.p.m. Condensed phosphates must first be hydrolysed to orthophosphates by boiling in acid soln. for 30 min.; Al can be masked with citrate. *Procedure*—Mix the acid soln. (containing 0·05 to 2 mg of Fe_2O_3) with 20% aq. citric acid (5 ml), 20% aq. Na_2SO_3 (5 ml) and 2% ethanolic quinol (5 ml), adjust to a pH of 4 to 5 and add a soln. of **I** in dil. HCl (10 ml). Heat on a water bath for 3 hr., cool, dilute to 100 ml and compare the extinction with that of a blank soln. in 1-cm cells with the use of filter Hg 546. A. R. ROGERS

900. Sampling and determination of hydrogen in steel. F. de Kazinczy. *Jernkontor. Ann.*, 1955, **139**, 309–325.—Melt the sample in a graphite crucible in a water-cooled quartz tube. Remove the water jacket and metallic film on the quartz tube and heat at 600° to 700° for 15 sec. to drive off adsorbed gas, of which 70 to 90% is H. The adsorbed H is significant when the H content of the sample or the content of the Mn or Cr is low. Determine the H content by diffusion through a lead membrane. The residual H in mild carbon steel, after vacuum extraction at 600°, has been determined by subsequent vacuum fusion. It has been found that residual H can be extracted by prolonged vacuum extraction at 600°, several days being required. Various methods of sampling are also described. CHEM. ABSTR.

901. Determination of magnesium in cast iron by the method of local [spectrographic] analysis. I. L. Mirkin and É. P. Rikman (Tul'sk Mechanical Inst.). *Zavod. Lab.*, 1956, **22** (8), 930–936.—Results of experiments on the determination of the distribution of Mg in cast iron by the method of local spectrographic analysis with high-frequency spark excitation are described. G. S. SMITH

902. Spectrochemical analysis of thermionic cathode nickel alloys by a graphite-to-metal arcing technique. E. K. Jaycox and B. E. Prescott (Bell Telephone Laboratories Inc., Murray Hill, N.J., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1544–1547.—A d.c. arc method has been devised to circumvent the disadvantages of the ASTM dry powder method

E–2, SM5–1, for determinations of Al, Co, Cr, Cu, Fe, Mg, Mn, Si and Ti in thermionic cathode nickel alloys, with an internal nickel standard. The sample is placed in the crater of a graphite cup (as anode) and excited in a 250-V d.c. arc at 16·5 amp. for 55 sec. For the determination of Al, Co, Cu, Fe, Mg, Mn and Ti in nickel over the concentration range 0·003 to 0·2%, the overall deviation was of the same order as that of the ASTM method, ±5·8%.

D. G. LLOYD

903. A direct micro-heterometric determination of palladium with dimethylglyoxime in excesses of foreign metals. M. Bobtelsky and B. Mayer (Hebrew Univ., Jerusalem, Israel). *Anal. Chim. Acta*, 1956, **15** (4), 373–378 (in English).—Palladous ions are pptd. quant. by 0·01 M dimethylglyoxime (in ethanol) at a pH $\geqslant 5$, the insol. complex dissolving only at a pH $\leqslant 11$. The influence of complexing agents and foreign cations on this reaction was studied heterometrically at 20°. Heterometric titrations of Pd can be made successfully in the presence of excess of conc. mineral acids, foreign metals (except coloured cations) and most complexing agents (sodium citrate, tartrate and pyrophosphate). In the presence of 50% of ethanol, acetone or dioxan the max. density and reaction-sensitivity are decreased. Large excesses of Cl⁻ can be tolerated. From 1 to 2 mg of Pd²⁺ can be titrated heterometrically with dimethylglyoxime in 20 ml of soln. containing >99% of Ca²⁺, Ba²⁺, Mg²⁺, Zn²⁺, Mn²⁺, Ni²⁺, Co²⁺, Al³⁺, Fe³⁺, Cr³⁺, Pb²⁺, Cd²⁺, Cu²⁺ and Hg²⁺. The titration time is ≃ 20 min. and the error is within ±3%. No filter should be used in the presence of Fe, Cr or Cu, and an excess of Na citrate should be added to mask the Fe.

W. J. BAKER

904. The determination of palladium and gold in igneous rocks by radioactivation analysis. E. A. Vincent and A. A. Smales (Dept. of Geol. and Min., Univ. Oxford). *Geochim. Cosmochim. Acta*, 1956, **9** (3), 154–160 (in English).—The methods of Goldberg and Brown (*Science*, 1949, **109**, 347; *Anal. Chem.*, 1950, **22**, 308) for the determination of Pd and Au in iron meteorites have been combined, somewhat modified and successfully applied to the determination of these elements in igneous rocks. Weighed samples (100 to 200 mg) of finely ground rock are enclosed in polyethylene tubing and simultaneously irradiated in a neutron pile with standards consisting of 10 mg of palladium foil and 5 mg of gold foil which have been weighed and similarly enclosed in polyethylene. After fusion with Na_2O_2, inactive palladium (10 mg) and gold (30 mg) foil are added as inactive carriers for the gravimetrically negligible atoms of the active isotopes. A chemical analysis is then performed to isolate the Pd and Au in a state of radiochemical purity. The recoveries of Pd as glyoxime and Au as metal (which need not be complete) are determined. The elements in the standards are similarly recovered in the same chemical forms as those from the rock samples. A direct comparison of the counting rates of the radioactivity isolated from sample and standards (corrected in each case for chemical yields) gives the ratio of the elements in the rock and in the standards. The limits of detection are set at 0·01 p.p.m. for Pd and 0·0005 p.p.m. for Au. The isotopes concerned are the 13·3-hr ¹⁰⁹Pd and the 2·7-day ¹⁹⁸Au. The results quoted show close agreement on duplicate samples and are of much the same order as those quoted in Goldschmidt's estimates for the upper lithosphere as a whole.

D. F. PHILLIPS

905. Separation and determination of palladium, selenium and mercury by reduction with ascorbic acid. M. T. Šušić and Z. B. Maksimović (Lab. of Phys. Chem., Inst. Nucl. Sci. "Boris Kidrich"). *Bull. Inst. Nucl. Sci., Belgrade*, 1956, **6**, 131–135.—Ascorbic acid reduces Pd, Se and Hg to the elementary form by reactions which are shown to be quant., and methods of determining these elements based on the iodimetric titration of excess of ascorbic acid have been developed; Pd^{II}, Se^{IV} and Hg^{II} are equiv., respectively, to 1, 2 and 1 mol. of ascorbic acid. The reactions are effected in acid medium.

S.C.I. ABSTR.

906. The colorimetric determination of iridium by o-dianisidine. S. S. Berman, F. E. Beamish and W. A. E. McBryde (Toronto Univ., Ontario, Canada). *Anal. Chim. Acta*, 1956, **15** (4), 363–366. —The spectrophotometric determination of Ir^{4+} can be made by adding a 0·5% (w/v) soln. of o-dianisidine in acetone to a buffered soln. (pH 4·7) of chloroiridate^{IV}, followed by addition of conc. HCl, and then measuring the max. extinction of the stable deep-purple coloration at 530 mμ. Beer's law is valid from 2 to 20 μg of Ir per ml. Other platinum metals, Ni, Cr, Cu and oxidising agents (Fe³⁺, Au³⁺, HNO₃, etc.) interfere. The sample soln. (in HCl) should be evaporated to dryness with conc. HNO₃ and 0·2% aq. NaCl (≃ 1 ml of each) and then twice with HCl. The residue is dissolved in H_2O (25 ml) and the colour is then developed in this soln. or in a 5-ml aliquot. The error is ±0·08%.

W. J. BAKER

907. Spectrographic analysis of a cracking catalyst. Tomio Okada, Shigeo Nakai and Tsunehide Kozuma (Maruzen Oil Co., Shimotsu, Wakayama Prefecture). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (5), 495–498.—Spectrographic analysis of trace amounts of Fe (2599·4 A), V (3184·0 A), Ni (3414·8 A), Cu (3247·5 A) and Na (3302·3 A) in a cracking catalyst, by the use of the wavelengths given, was studied with reference to the preparation of the sample electrode and the choice of internal standard. A new type of electrode was devised. A hole (diameter 3 mm) is pierced through the centre of a carbon rod. A glass rod (diameter 3 mm) is inserted into the hole so that its end supports the solid sample near the end of the carbon rod. This glass is moved up and down with a holder equipped with a gear system, to keep the upper surface of the sample at the opening of the electrode. An intermittent arc (220 V, 5 A, interruption factor 1/4, period 1 sec., exposure 20 sec.) is struck between this and pure carbon. Silicon (2532·4 A) is used as an internal standard. The standard deviation is ≃ ±4% for 0·01 to 0·1% of Fe, 0·01 to 0·8% of Ni and 0·001 to 0·05% of Cu, ≃ ±7% for 0·01 to 0·4% of V and ≃ ±15% for 0·01 to 0·6% of Na.

K. SAITO

See also Abstracts 783, 784, 785, 788, 789, 794, 795, 944, 956, 990, 1076, 1083, 1084, 1085, 1090, 1091.

3.—ORGANIC ANALYSIS

908. Some ultra-micro methods of quantitative organic elementary analysis. W. J. Kirsten (Inst. Med. Chemie der Univ., Uppsala, Sweden). *Mikrochim. Acta*, 1956, (4–6), 836–842.—A description of ultra-micro determinations of S, N, C and H is given.

M. F. C. LADD

909. Application of the thermal decomposition products of silver permanganate in organic elementary analysis. J. Körbl (Forschungsinst. für Pharmazie und Biochem., Prague, Czechoslovakia). *Mikrochim. Acta*, 1956, (11), 1705–1721.—The uses of the products of the thermal decomposition of $AgMnO_4$ in organic analysis are summarised. Topics discussed include the determination of C and H, with a $AgMnO_2$ catalyst, with modifications for analysing volatile and Si-containing samples; the possibility of determining S; and the detection of C, H, N, Cl, Br, I, P, As and Hg.

A. R. ROGERS

910. The use of silver permanganate in analysis. VI. A simple and rapid method for the detection of the elements in organic compounds. J. Körbl and R. Přibil (Pharm. & Biochem. Res. Inst., Czech. Acad. Sci., Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1956, **21** (4), 955–959 (in German). —A simple micro-method for the detection of C, H, Hg, N, Cl, Br, I, S, P and As in organic compounds is described. From 0·1 to 1 mg of the compound is gently fused in a special tube with the decomposition product of $AgMnO_4$; H is detected by the formation of dew in the cold part of the tube; C by the formation of CO_2 which is detected by $Ba(OH)_2$; Hg condenses on the cold part of the tube and is detected by dithizone; the halogens give silver halides which are leached out differentially (aq. NH_3 dissolves AgCl and AgBr, 30% $AgNO_3$ dissolves AgI); S gives silver and manganese sulphates which are detected with $BaCl_2$; P and As give phosphate and arsenate which are detected with molybdate; and N gives nitrate and nitric acid which are detected with diphenylamine. The full procedure can be carried out in a few minutes.

P. S. STROSS

911. Some further deliberations on the rapid combustion procedure. G. Ingram (Research Lab., Courtaulds Ltd., Maidenhead, Berks., England). *Mikrochim. Acta*, 1956, (4–6), 877–898 (in English). —By using the baffle-chamber type of combustion tube a superior method of analysis can be carried out in a very short period of time. For C and H, the sample is decomposed at 900° in a turbulent flow of O; the accuracy attained is $\pm 0\cdot 05\%$ for C and $\pm 0\cdot 1\%$ for H. Oxides of nitrogen are retained by MnO_2. Halogens are determined potentiometrically with $AgNO_3$. The combustion products of sulphur are retained by hot silver wire, the Ag_2SO_4 formed being then titrated potentiometrically with NaCl soln.

M. F. C. LADD

912. The use of copper in micro-determinations of carbon and hydrogen. G. Kainz, A. Resch and F. Schöller (Chem. Lab., Univ. Vienna). *Mikrochim. Acta*, 1956, (4–6), 850–857.—An apparatus previously described (*Mikrochem.*, 1950, **35**, 569; 1952, **39**, 166) for series micro-determinations of C and H has been simplified. Oxides of nitrogen are reduced by copper and the preparation of an active surface of copper is described. The duration of the series analysis is 30 min. M. F. C. LADD

913. New apparatus for the semi-micro and micro-determination of active hydrogen in organic compounds. M. Souček (Inst. Anal. Chem., High-School of Chem. Technol., Pardubice, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 323–325.—The gas, released from a compound by reaction with Grignard reagent or $LiAlH_4$, depresses the level of liquid in one arm of a manometer. A stoppered burette is connected to the reaction vessel and is filled with the liquid, which is run out until the levels of the liquid in the manometer are equalised. From the volume of liquid run out, the hydrogen content of the compound can be calculated. J. ZÝKA

914. Crystallised anhydro-iodic acid as oxidising agent in the direct micro-estimation of oxygen in organic substances. J. Unterzaucher (Org.-anal. Lab. der Farbenfabriken Bayer A.-G., Leverkusen). *Mikrochim. Acta*, 1956, (4–6), 822–835.—In the preparation of I_2O_5, anhydro-iodic acid ($HIO_3 . I_2O_5$) is obtained below 196° and I_2O_5 above 196°. Pure anhydro-iodic acid was prepared by crystallising I_2O_5 from boiling 60% HNO_3. In contrast to I_2O_5, anhydro-iodic acid does not react with H. Carbon monoxide reacts with anhydro-iodic acid according to the equation—

$$15CO + 2(HIO_3 . I_2O_5) \rightarrow 15CO_2 + 3I_2 + H_2O.$$

Micro-determinations of O were carried out on several compounds by determining the CO in the thermal decomposition products. With oxygen-free substances, *e.g.*, carbazole, anhydro-iodic acid gave oxygen values of 0·03 to 0·04%, whereas I_2O_5 gave values of about 1·6%. Oxygen-containing compounds free from H were determined equally well by both methods. In other instances, determinations with anhydro-iodic acid gave oxygen values which were lower and in better agreement with the theoretical values than did I_2O_5. M. F. C. LADD

915. Contribution to the Unterzaucher process for the micro-analytical determination of oxygen. W. Schöniger (Mikroanal. Lab. der pharm. Abteilung der Sandoz A.-G., Basel, Switzerland). *Mikrochim. Acta*, 1956, (4–6), 863–868.—Graphitisation of the contact-tube filling in the Unterzaucher process for the determination of O has been shown to lead to low results. It is recommended that re-charging of the tube should be made at three-monthly intervals. M. F. C. LADD

916. Micro-determination of halogens in organic compounds. R. Lévy (Lab. Municipal de Paris, Section de Microanalyse). *Mikrochim. Acta*, 1956, (4–6), 906–924.—An earlier method for the micro-determination of Cl⁻ and Br⁻ (*cf. Brit. Abstr. C*, 1952, 399) is extended to a mixture of these two anions. The organic compound is mineralised with Na_2O_2 and the halogens are determined potentiometrically with $AgNO_3$. Two samples are used; on the first the sum of Br⁻ and Cl⁻ is determined and on the second only Br⁻ is found, with 0·01 N and 0·002 N solutions of $AgNO_3$, respectively. The mean absolute error is $\pm 0\cdot 15\%$ for Br and $\pm 0\cdot 20\%$ for Cl. M. F. C. LADD

917. Studies in organic elementary analysis. XVI. Micro-determination of halogens in organic compounds. Tetsuo Mitsui and Hiromu Sato (Coll. of Agric., Univ. Kyoto, Japan). *Mikrochim. Acta*, 1956, (11), 1603–1616 (in English).—The method of Safford and Stragand (*Anal. Chem.*, 1951, **23**, 520) for the micro-determination of halogens has been modified to improve its precision. The silver-gauze absorbent has been replaced by electrolytic silver wool, which presents a much greater surface and absorbs 40 or 50 times as much halogen. A special absorption funnel has been designed to facilitate handling and weighing of the absorbent. The operating range of the silver wool is wide (350° to 450°). The modified method compares favourably in accuracy, precision (coefficient of variation $\simeq 0\cdot 12\%$) and simplicity with established methods.

A. R. ROGERS

918. Micro-estimation of fluorine in organic substances. G. Kainz and F. Schöller (Chem. Lab., Univ. Vienna). *Mikrochim. Acta*, 1956, (4–6), 843–849.—The organic fluorine-containing compound is decomposed by heating with K in a glass tube. If the tube is pre-treated with hot HCl no blank correction is necessary. The fluoride solution is buffered to a pH of 3·3 with glycerol - $HClO_4$ and titrated with $Th(NO_3)_4$, with alizarin red S as indicator. The limits of error are $\pm 0.4\%$.
M. F. C. LADD

919. The micro-analysis of fluorine-containing organic compounds. IV. Determination of fluorine in presence of phosphorus or arsenic. R. Belcher and A. M. G. Macdonald (Univ. Birmingham, England). *Mikrochim. Acta*, 1956, (4–6), 899–905 (in English).—The organic compound is decomposed by fusion with Na in a nickel bomb. Arsenate and phosphate are removed with zinc carbonate, and fluoride is determined by addition of an excess of $Th(NO_3)_4$ soln. and back-titrating with NaF soln., with alizarin red S as indicator. Determinations of F^- were carried out, *e.g.*, with a mixture of 3-trifluoromethylbenzoic acid and triphenylphosphine; an average error of 0·26% and a maximum error of 0·5% are quoted.
M. F. C. LADD

920. Micro-analysis of perfluoro compounds. C. A. Rush, S. S. Cruikshank and E. H. J. Rhodes (Chem. and Radiological Lab., Army Chem. Center, Md., U.S.A.). *Mikrochim. Acta*, 1956, (4–6), 858–862 (in English).—The elements P, Cl, Br, I and S are determined by the Carius digestion method followed by standard gravimetric analyses. Nitrogen is determined by a conventional micro-Dumas method. Carbon and H are determined by combustion in O, and F is determined by reaction with Na in a conventional sodium peroxide bomb; the NaF formed is titrated with $Th(NO_3)_4$. A satisfactory determination of O has not been developed. Perfluorodibutyl ether, $(C_4F_9)_2O$, is the standard compound used for checking the apparatus. M. F. C. LADD

921. New method for determination of chlorine and fluorine in gaseous and liquid fluorinated hydrocarbons. V. Crespi and F. Cevolani ("G. Donegani" Research Inst., Soc. Montecatini, Novara, Italy). *Chim. e Ind.*, 1956, **38** (7), 583–587.—For the determination of F, the sample is first decomposed on a mixture of Na_2CO_3 and SiO_2 at 750° (apparatus described), which fixes F as NaF and Na_2SiF_6. The determination is accomplished by solution in water, pptn. of F^- by Ce^{3+} and titration of excess of Ce^{3+} with a sequestering agent. Chlorine is determined by the Volhard method. The method is both precise and convenient. C. A. FINCH

922. New method for the determination of chlorine in organic substances. L. Vettori and M. Restum (Inst. Quím. Agrícola, Rio de Janeiro). *Bol. Inst. Quím. Agríc.*, 1955, No. 41, 16 pp.—The substance (0·1 to 0·5 g) is heated in a combustion tube in a current of air, where it is vaporised and partly decomposed. The vapours are then passed through a flame of illuminating gas burning in a specially constructed burner at the end of the tube in which all the Cl is converted into HCl. The products of combustion are then pumped into a Kjeldahl flask containing an alkaline soln. of sodium arsenite. The Cl^- are then determined electrometrically or volumetrically with $AgNO_3$ and SCN^-. Results are reported on dicophane, γ-hexachloro*cyclo*hexane, hexachloroethane and various commercial insecticides. L. A. O'NEILL

923. Application of the Eschka method to the determination of chlorine and total sulphur in gaseous compounds. V. Crespi and F. Cevolani ("G. Donegani" Res. Inst., Soc. Montecatini, Novara, Italy). *Chim. e Ind.*, 1956, **38** (7), 580–582. —The apparatus employed and the results of several determinations are reported. C. A. FINCH

924. The rapid micro-analytical determination of halogens and sulphur in organic compounds. W. Schöniger (Mikroanal. Lab. pharm. Abteilung der Sandoz A.-G., Basel, Switzerland). *Mikrochim. Acta*, 1956, (4–6), 869–876.—In the combustion method described, sulphur, as H_2SO_4, is titrated with $BaCl_2$ and EDTA (disodium salt); Br with $Na_2S_2O_3$; F with ceric chloride; Cl and I by standard procedures. M. F. C. LADD

925. Organic quantitative analysis. VI. Micro-determination of sulphur in organic compounds. M. Večeřa (Res. Inst. of Org. Synthesis, Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 308–311.—In a previous communication of this series (*Anal. Abstr.*, 1955, **2**, 1238) a new method for the determination of sulphur in organic compounds was described. In order to increase the reliability of this method, the effects on the procedure of the following were studied—the velocity of the combustion, temperature, the extraction of Ag_2SO_4 and the chlorine content of the compound to be analysed.
J. ZÝKA

926. The estimation of small quantities of sulphur. F. Čůta, M. Hejtmánek and Z. Kučera (Tech. High School for Chem., Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1956, **21** (4), 886–889 (in German).—Sulphur-containing compounds such as NH_4SCN, thiourea, cystine, $Na_2S_2O_3$ and allyl *iso*thiocyanate are reduced by Al in boiling HCl to H_2S, which is distilled into a soln. of a lead salt and the colour is determined absorptiometrically. To a soln. containing $\not> 40$ μg of combined S in a distillation apparatus add aluminium foil, displace the air by H and add HCl from a separating funnel. Boil, and collect the distillate in a flask containing dil. aq. NH_3 (20 ml) and absorption soln. [5 g of $Pb(NO_3)_2$ in 50 ml of warm water and 20 g of citric acid, diluted to 100 ml and filtered] (1 ml). Measure the extinction (Ilford 601 filter) in a 4-cm cell and compare with a calibration curve obtained by reducing a pure sample of the same compound that is being assayed. From 3 to 40 μg of S can be estimated with an accuracy within $\pm 7\%$.
P. S. STROSS

927. Micro-analytical determination of various sulphur compounds in gases and solids. H. Stratmann (Kohlenstoffbiologischen Forschungsstatione. V. Essen). *Mikrochim. Acta*, 1956, (4–6), 1031–1037. —Sulphur-containing compounds are reduced by H to H_2S, which is then determined by the molybdenum blue reaction; 0·1 μg can be determined quantitatively. M. F. C. LADD

928. Identification of organic compounds. X. Identification of sulphides. M. Večeřa and J. Petránek (Res. Inst. of Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 240–245.—The optimal conditions for the preparation of SS-dialkyl-N-*p*-toluenesulphonylsulphilimines as deriv. suitable for identification of thioethers, with chloramine T as reagent, have been found. *Procedure*—Mix 0·005 mole of chloramine T, dissolved in methanol (10 ml), with a solution of

0·005 mole of sulphide, dissolved in 10 ml of the same solvent. Remove the solvent by distillation, wash the residue with $2N$ NaOH (5 ml) and with water (20 ml). Dissolve the product in toluene (10 ml) on a boiling-water bath, filter, add *cyclo*-hexanol till the first turbidity appears and leave the product to crystallise. Sulphilimines of 23 sulphides were prepared and their identification by their eutectic points with different compounds on Kofler's block is described. The usefulness of this method for the identification of thioethers is discussed. J. ZÝKA

929. **Organosilicon compounds. VI. The determination of silicon in organosilicon compounds.** Z. Šír and R. Komers (Chem. Inst. Czech. Acad. Sci., Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1956, **21** (4), 873–879 (in German).—The method is suitable for the determination of Si in esters of orthosilicic acid, arylsilanes, arylalkylsilanes, polysiloxanes, alkylsilanes and alkyl- and aryl-halogenosilanes. The determination of Si is carried out acidimetrically. A mixture of oleum and fuming HNO_3 is used for the oxidation, which is followed by ignition. Four different techniques are used according to the volatility and ease of oxidation of the compound. Non-volatile compounds are weighed directly into a platinum crucible to which the oxidants are added. Alkylsilanes are weighed into a crucible, cooled in solid CO_2, containing the oxidants. Arylhalogenosilanes are oxidised in H_2SO_4 - HNO_3 containing a little H_2O, and alkylhalogenosilanes are weighed into a platinum crucible containing pyridine cooled in solid CO_2, and the oxidants are added to this. *Procedure*—Heat the crucible gently on a sand bath, then more strongly until the contents are ignited. Dissolve the residue in the minimum amount of water containing Na_2CO_3 or K_2CO_3 (0·5 to 1 g), transfer to a flask, add excess of dil. HCl (1 + 1) and boil to remove CO_2. Add 10 drops of a mixture of alcoholic methyl red soln. (0·1%) and aq. bromocresol green soln. (0·1%), exactly neutralise with NaOH, keeping the vol. below 50 ml, saturate with KCl or KNO_3, add neutral NH_4F soln. (1%) (10 ml) and exactly 20 ml of 0·1 N HCl. Back-titrate the excess of acid with 0·1 N NaOH until the colour changes to green. The method has been applied to some hundreds of organic compounds, details of several of which are given. P. S. STROSS

930. **Volumetric micro-determination of arsenic in organic compounds by means of magnesium fusion.** M. Jureček and J. Jeník (Tech. High School for Chem., Pardubice, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1956, **21** (4), 890–893 (in German). —A rapid and widely applicable micro-method for the determination of organically combined As is described. The As is converted by fusion with Mg into magnesium arsenide, this is decomposed by H_2SO_4 to arsine, which is oxidised by Br and determined iodimetrically. *Procedure*—Fuse 2 to 4 mg of the compound with magnesium powder in a glass tube. Fracture the tube and transfer to a distillation apparatus; displace the air by N or CO_2 and add H_2SO_4 (40%) (10 ml) from a separating funnel; after the interaction, sweep the AsH_3 formed into an absorption tube containing bromine water (1%) (10 ml). To this soln. add sodium acetate (1 g) and formic acid (3 drops) and, when colourless, conc. HCl (15 ml exactly) followed by KI soln. (4%) (2 ml). Mix and set aside in the dark for 15 to 20 min. and titrate the liberated I with 0·01 N $Na_2S_2O_3$, with starch as indicator. Repeat

the determination omitting the sample. Each determination takes 60 to 80 min.; the limits of error are ±0·3%. P. S. STROSS

931. **General determination of acetyl.** A. Chaney and M. L. Wolfrom (Ohio State Univ., Columbus, Ohio, U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1614–1615.—The method is a modification of the Ost technique (*Z. angew. Chem.*, 1906, **19**, 993), and involves distillation of the sample from toluene-*p*-sulphonic acid. The acetyl content is measured by titration of the distillate with standard alkali. Results of the analysis of about 20 compounds are listed. G. P. COOK

932. **Recent advances in the application of micromethods to the identification of organic compounds.** N. D. Cheronis (Brooklyn College, Brooklyn, N.Y., U.S.A.). *Mikrochim. Acta*, 1956, (4–6), 925–947 (in English).—The concept of "proof of identity" is critically reviewed. The author believes that, besides colour reactions, data on at least two constants should be available, *e.g.*, m.p. of a derivative of the compound. Procedures are given for preparation of derivatives in milligram quantities. With microgram quantities the preparation of a derivative and characterisation by R_F values are illustrated with the fatty acids: the 2 : 4-dinitrophenylhydrazide derivatives of the fatty acids were prepared, their chromatographic properties studied and satisfactory resolution was obtained. The use of some new reagents, *e.g.*, biphenyl tetrazolium violet (2 : 5-diphenyl-3-*o*-biphenyltetrazolium chloride), for the micro-detection of reducing sugars is described. M. F. C. LADD

933. **Trace unsaturation of hydrocarbons by coulometric titration.** F. A. Leisey and J. F. Grutsch [Standard Oil Co. (Indiana), Whiting, Ind., U.S.A.]. *Anal. Chem.*, 1956, **28** (10), 1553–1555.—Bromine, generated in the titration cell by oxidation of Br⁻ in the electrolyte, reacts with the unsaturated compounds in the sample. The end-point is detected by a reference current and the generation time determines the bromine index of the sample. At the end-point, a small excess of Br increases the current flow to the detection electrodes and causes a relay circuit to stop the generation current and electrical timer. Agreement to within 5% of theoretical values was obtained over the bromine-index range of 0 to 180. G. P. COOK

934. **Industrial analysis by optical methods. IV. Analysis of impurities in synthetic methanol by the Raman spectrum.** Yoichiro Mashiko, Shinnosuke Saeki and Hiroshi Tomiya (Gov. Chem. Ind. Res. Inst., Yoyogi P.O., Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (2), 239–243.—Crude methanol is separated into three fractions and each of them is submitted to the measurement of their Raman spectra for qual. analysis. The first fraction (boiling range 15° to 60°) contains dimethyl ether (**I**) and methyl formate (**II**) as main constituents. The second fraction consists mainly of methanol. The third fraction (boiling range 70° to 138°) was again split into 20 fractions (mainly higher alcohols) and the main components were identified by their Raman spectra.

V. Infra-red analysis of the lower boiling by-products of synthetic methanol. Yoichiro Mashiko, Shinnosuke Saeki and Toshio Yoshimoto. *Ibid.*, 1956, **59** (2), 243–250.—The above-mentioned first fraction was submitted to i.r. absorption measurement for closer studies of the main components.

This fraction was further split into 40 fractions and their i.r. spectra recorded. Besides **I** and **II**, several compounds, including methylal, *iso*butyl methyl ether, methyl acetate, dimethylacetal, *n*-heptane and *n*-octane were identified.

VI. Infra-red analysis of the higher boiling by-products in synthetic methanol. Shinnosuke Saeki, Toshio Yoshimoto and Yoichiro Mashiko. *Ibid.*, 1956, **59** (2), 250–259.—Similar experiments were carried out with the third fraction; it was split into 20 fractions and examined by i.r. spectrophotometry. The bands of ethanol, *n*-propanol, *iso*butyl alcohol, *iso*amyl alcohol, *sec.*-butyl alcohol, *n*-pentanol, *n*-hexanol and 3-pentanol were identified. In fractions of b.p. > 130°, distinctive absorption bands due to carbonyl groups were observed.　　　　　　　　　　　　　K. SAITO

935. A contribution to the determination of methanol in aqueous solutions of formaldehyde. A. Mervartová and J. Chytílek (Research Inst.Plastics, Gottwaldov, Czechoslovakia). *Chem. Průmysl*, 1955, **5** (10), 435–436.—The Vieböck - Schwappach modification of Zeisel's method for determining methoxyl groups has been proved to be suitable for the determination of methanol in aqueous solutions of formaldehyde. For the determination of solutions containing more than 20% of methanol, the Fierz-David apparatus must be used.

J. ZÝKA

936. Determination of vapours of diethyl ether in a mixture of gases. O. Růžička (Research Inst. Synth. Rubber, Gottwaldov, Czechoslovakia). *Chem. Průmysl*, 1955, **5** (5), 218.—A suspension of ferrocyanic acid in dil. H_2SO_4 has been found to be a suitable absorbent for diethyl ether vapour in gases (ethane, ethylene, butadiene) resulting from the thermal catalytic decomposition of ethanol. The determination can be carried out with the Orsat apparatus. In the determination of up to 10% of ether vapour in gases, the error did not exceed ±1·5%. The presence of propenes, butenes and butadiene does not cause interference.　　J. ZÝKA

937. Slide test for fructose and other ketohexoses. R. K. Maurmeyer, E. M. Livingston and R. Michaels (Chem. Dept., Brooklyn Coll., Brooklyn, N.Y., U.S.A.). *Mikrochim. Acta*, 1956, (11), 1600–1602 (in English).—Ketohexoses or sugars that hydrolyse to ketohexoses give a green colour when heated for 1 min. with a soln. of phenol (13 g) in glacial acetic acid (74 ml) and conc. H_2SO_4 (13 ml); aldohexoses give a yellow or red - brown colour which develops more slowly (*cf.* Maurmeyer *et al.*, *J. Biol. Chem.*, 1950, **185**, 347). The test can be carried out on a microscope slide with quantities of ≃ 1 μg. Fructose can be detected by this method in the presence of reasonably large amounts of other reducing monosaccharides.　　　　A. R. ROGERS

938. The anthrone reagent. Application to determination of heptuloses. L. P. Zill (Oak Ridge Nat. Lab., Oak Ridge, Tenn., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1577–1580.—Evidence is presented that implicates NO_3^- in the instability of the anthrone reagent. Increased stability of the reagent is obtained by the use of low anthrone and acid concn. and by the addition of thiourea, as proposed by Roe (*Anal. Abstr.*, 1955, **2**, 1614). The reagent is applicable to the determination of heptuloses, an absorption max. at 515 mμ being suitable for quantitative determinations. Max. colour development at this wavelength is obtained

with 20 min. heating at 100°. Linear absorbance with concn. is obtained over the 20 to 200-μg range.

G. P. COOK

939. The use of thiocarbohydrazones in functional organic analysis. C. Duval and N. Dat Xuong (Lab. Recherches Micro-analytiques, 11 rue Pierre Curie, Paris). *Mikrochim. Acta*, 1956, (4–6), 747–749.—Thiocarbohydrazide $[S:C(NH·NH_2)_2]$ reacts quantitatively with aldehydes and some ketones to form yellow or red crystalline derivatives. Thiocarbohydrazide (1 mole) in 50% aq. alcohol and the aldehyde or ketone (2 moles) in 96% ethanol are mixed and maintained at 50° for approx. 30 min. The ppt. is washed with water and finally with 50% aq. alcohol. The regions of stability of the ppt. have been determined thermogravimetrically, and the melting-points and safe drying temperatures for the derivatives of a number of aldehydes and ketones are listed. M. F. C. LADD

940. Spectrophotometric determination of traces of formaldehyde. P. W. West and B. Sen (Coates Chem. Lab., Louisiana State Univ., Baton Rouge, U.S.A.). *Z. anal. Chem.*, 1956, **153** (3), 177–183 (in English).—The rate of production and intensity of the purple - violet colour obtained by the interaction of formaldehyde (**I**) and chromotropic acid (**II**) in H_2SO_4 soln. depend on the concn. of acid during the colour development, the temp. and the amount of **I**. The colour produced by the recommended procedure obeys Beer's law in the concn. range 0·05 to 2·0 p.p.m. of **I**. Acraldehyde interferes, but the interference can be reduced by using a fivefold excess of **II** reagent. 2:7-Dihydroxynaphthalene can replace **II**, but the intensity of colour (which has $\lambda_{max.} = 540$ mμ) is more dependent on the acid concn. *Procedure*—Mix the aq. soln. of **I** with a 1% soln. of Na chromotropate in conc. H_2SO_4 (equal vol.), immediately add sufficiently conc. H_2SO_4 to give an acid concn. of ≮86%, mix, dilute cautiously with H_2O, cool and dilute to volume. Measure the extinction at 570 mμ in 1-cm cells.　　　　　A. R. ROGERS

941. Determination of small quantities of acetaldehyde in aqueous solutions. N. R. M. Impey and E. T. Verdier (Rhodes Univ., Grahamstown, S. Africa). *J. S. Afr. Chem. Inst.*, 1956, **9** (1), 51–54. —Polarographic and volumetric methods for determination of acetaldehyde in solution are reviewed and a volumetric procedure is developed. The sample (2 ml) is neutralised (if necessary) with 0·1 *N* NaOH, 25% aq. $NaHSO_3$ (20 ml) is added and allowed to stand for ≃ 1·5 hr., then cooled in ice for 20 to 30 min. Excess of $NaHSO_3$ is removed by addition of iodine soln. (20 g of I and 37 g of KI per litre) to a faint-blue end-point with starch. The soln. is then cooled again in ice for 5 min., saturated aq. $NaHCO_3$ (15 ml) is added and the mixture is titrated with 0·008 *N* KI, adding 2 ml of 10% aq. Na_2CO_3 near the end-point to ensure completion of reaction. A correction factor was required. The standard deviation was 1·5%.　　　　　　　　　　　S.C.I. ABSTR.

942. The detection of unsaturated fatty acids on paper chromatograms. J. Fries, A. Holasek and H. Lieb (Med.-chem. Inst., Univ. Graz, Austria). *Mikrochim. Acta*, 1956, (11), 1722-1726.—A mixture of fatty acids can be separated by chromatography on paper that has been impregnated with liquid paraffin; the unsaturated fatty acids are revealed by exposing to ozone for 20 to 60 min., then spraying

with Schiff reagent. Liquid paraffin can be freed from unsaturated compounds by shaking successively with oleum, conc. H_2SO_4, aq. $KMnO_4$ and conc. H_2SO_4, then washing with aq. NaOH and H_2O.

A. R. ROGERS

943. Near-infra-red spectra of fatty acids and some related substances. R. T. Holman and P. R. Edmondson (Univ.of Minn., Austin, Minn., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1533–1538.—The absorption spectra of 63 fatty acids and related substances have been studied between 0·9 and 3·0 μ. By means of these spectra, *cis* configuration, terminal double-bonds, hydroxyl, amino, hydroperoxide, acid, methylene and methyl groups, and methyl and ethyl esters can be distinguished.

G. P. COOK

944. A new colour reaction for oxalic dihydrazide. G. Nilsson (Inst. für Byggnadsteknik, Kungl. Tekniska Högskolan, Stockholm, Sweden). *Z. anal. Chem.*, 1956, **153** (3), 161–165.—Oxalic dihydrazide (**I**) gives a violet colour with ammoniacal copper salts in the presence of an excess of certain aliphatic aldehydes. Some other hydrazides give much less intense colours. The reaction can be used for the detection of 0·1 p.p.m. of Cu or 10 p.p.m. of **I** (or of ethyl oxalate or hydrazine hydrate). With the higher aldehydes, the coloured product can be extracted by *n*-butanol. Cyanide and sulphite prevent the reaction, and thiocyanate, thiosulphate, tartrate, pyrophosphate and citrate should preferably be absent. A. R. ROGERS

945. Spectrophotometric determination of microgram quantities of ethyl acetoacetate. F. N. McMillan (Eglin Air Force Base, Fla., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1532–1533.—The method is based on the u.v. absorption of ethyl acetoacetate in aq. NaOH at 272·5 mμ. Beer's law is obeyed over the concn. range of 1 to 7 p.p.m. and the standard deviation over this range is $\pm$0·04 p.p.m. G. P. COOK

946. The spectrophotometric determination of secondary aliphatic amines. S. J. Clark and D. J. Morgan (British Nylon Spinners Ltd., Pontypool, Mon., England). *Mikrochim. Acta*, 1956, (4–6), 966–976 (in English).—The reaction with HNO_2 has been studied for several amines and found to be rapid and reproducible. Amines may be treated with HNO_2 and determined spectrophotometrically; amounts in the range 0·1 to 1·0 milli-equiv. are found to within approximately 3%. Primary aliphatic amines interfere and a correction can be applied; tertiary amines may be present in moderate quantities without disadvantage. M. F. C. LADD

947. Photometric determination of chlorobenzene in benzene. V. Šedivec (Inst. for Ind. Hyg. and Occupational Diseases, Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1956, **21** (4), 965–969 (in German).—A method for the estimation of chlorobenzene (**I**) in benzene is described, based on the nitration of **I** in glacial acetic acid and the colorimetric determination of the formed 1-chloro-2:4-dinitrobenzene. Dilute a 0·05 to 0·2-g sample to 10 ml with glacial acetic acid. To 0·2 ml add nitration mixture (fuming HNO_3 - conc. H_2SO_4, 1:2 by vol.) (2 ml), mix and allow to stand for 15 min.; add H_2O (15 ml) and pyridine (10 ml). Heat for 25 min. at 90° to 95°, cool, transfer to a separator, add NaOH (40%) (10 ml), shake, and separate the coloured pyridine layer. To this add NaOH (4%)

(1 ml) and dilute to 25 ml with water. Determine the extinction, with a green filter, and compare with a calibration curve. Xylenes interfere if present in quantities comparable with that of **I**.

P. S. STROSS

948. Chemical determination of xylene isomers. Akito Kobayashi, Shizuo Nagahama and Saburo Akiyoshi (Dept. of Applied Chem., Faculty of Eng., Kyushu Univ., Hakozaki, Fukuoka). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (2), 179–182.— Reichel's nitration method for the determination of xylene isomers (*ChemZtg*, 1931, **55**, 744; *Chem. Abstr.*, 1943, **37**, 2305) was re-examined with reference to the nitration of the *p*-isomer. Nitration of *p*-xylene to yield 2:3:5-trinitro-*p*-xylene proceeds to only 70% of the theoretical value at 110°. The empirical calibration curve should therefore be revised. *o*-Xylene is also nitrated under the given conditions to produce 3:6-dinitro-*o*-xylene. Since the latter is sparingly sol. in acetone or ethanol, the amount of acetone should be increased to 200 ml in the presence of > 3 g of *o*-xylene in 5 ml of the sample. The error of the results for *m*- and *p*-isomers is 1 and 2%, respectively. K. SAITO

949. Analysis of error in the rapid determination of anthracene by the diene method. I. The chlorobenzene partition method and the xylene distillation method. Eiichi Funakubo, Yutaro Matsumoto and Hiroshi Taniguchi (Faculty of Eng., Osaka Univ., Miyakojima-ku, Osaka). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (4), 422–429. —Analytical results for the determination of anthracene (**I**) with maleic anhydride (**II**) were statistically analysed and its reliability is discussed. The modified chlorobenzene partition method with an empirical correction factor (Funakubo *et al.*, *Ibid.*, 1954, **57**, 563) has advantages since it requires neither preliminary experiments nor the prep. of specially pure **II**. The error is < $\pm$0·5% whatever the composition of the sample. The main source of error for the xylene distillation method occurs in the removal of the xylene; a significant part of the addition compound between **I** and **II** is converted into an unknown substance during the distillation, which consumes alkali on the titration of the excess of maleic acid (Funakubo *et al.*, *Ibid.*, 1954, **57**, 557). This method is suitable only for 40 to 100% of **I**. For the application of the Schmidt method (*BrennstChemie*, 1952, **33**, 176), the purity must be > 99·7%.

II. Examination of the "JIS" (Japan Industrial Standard) method. Eiichi Funakubo and Yutaro Matsumoto. *Ibid.*, 1956, **59** (4), 429–436.—The statistical analysis of error in determination of **I** by the "JIS" methods, the chlorobenzene method without correction and the Schmidt method, was similarly made. It is proposed that the authors' empirical factor should be used in the calculation for the chlorobenzene method. Kisfaludy's dioxan method (*Magyar Kém. Foly.*, 1952, **58**, 312) was also examined, but was found to be no better than the above-mentioned methods. K. SAITO

950. High-frequency titrimetry. I. Titration of phenols and phenolic mixtures in non-aqueous media. K. J. Karrman and G. Johansson (Anal. Chem. Dept., Univ. Lund, Sweden). *Mikrochim. Acta*, 1956, (11), 1573–1584 (in English).—High-frequency titrations have been carried out by using a crystal-controlled oscillator at 5 megacycles per second. The change in high-frequency conductance during the titration is measured by tuning to resonance by

means of a variable condenser in parallel with the titration cell in the anode circuit and determining the grid bias with a valve voltmeter after each addition of titrant. Phenol, *o*-, *m*- and *p*-cresol, 1- and 2-naphthol, 2-retenol (1-methyl-7-*iso*propyl-2-phenanthrol), *o*-, *m*- and *p*-dihydroxybenzene, pyrogallol and phloroglucinol (0·5 to 5 mg) were dissolved in benzene - methanol (2: 1) and titrated with 0·03 *N* K methoxide in benzene - methanol (10: 1). Each hydroxyl group in the molecule gives rise to a break in the titration curve. When a mixture of phenols is titrated, each component causes a break, in order of decreasing dissociation constant.   A. R. ROGERS

951. **Determination of 4-methoxyphenol in 2-*tert.*-butyl-4-methoxyphenol by partition chromatography and ultra-violet spectroscopy.** D. R. Long and R. W. Neuzil (Universal Oil Products Co., Des Plaines, Ill., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1547–1549.—The 4-methoxyphenol (**I**) is separated from 2-*tert.*-butyl-4-methoxyphenol (**II**) by liquid - liquid partition chromatography. The stationary phase consists of a silica-gel column saturated with water and the mobile phase consists of water-saturated CCl_4. The fractions are measured for **I** and **II** by u.v. spectroscopy at 294 and 292 mμ and, after separation, the final concn. of **I** is measured at the former wavelength. As little as 0·03% of **I** in **II** can be accurately determined.
                                    G. P. COOK

952. **Electrophoresis. II. Paper electrophoresis of organic acids and phenols.** H. Berbalk and O. Schier (Inst. organ. Chemie, Tech. Hochschule, Vienna). *Monatsh.*, 1955, **86**, 146–153.—Paper electrophoresis at pH 2 of many organic acids in aqueous solution was carried out and it was found that the rate of movement was related to the dissociation constant. A combination of electrophoresis and chromatography is described for the determination of an unknown acid mixture. Electrophoresis for 10 to 30 min. at 75 V per cm was followed by chromatography for 16 to 24 hr.   D. P. FELIX

953. **Colour reaction of 1-chloro-2:4-dinitrobenzene with pyridine.** V. Šedivec (Inst. for Ind. Hyg. and Occupational Diseases, Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1956, **21** (4), 820–824 (in German).—The method of Grabovicz (*Med. Pracy*, 1954, **5**, 247) was studied to find the optimum conditions, and some modifications of the temperature and time factors were made. The use of water as solvent instead of ethanol overcomes the interference of dinitrobenzene and dinitrotoluene but necessitates the use of large quantities of pyridine. *Procedure*—Dilute a soln. of 1-chloro-2:4-dinitrobenzene (0 to 200 μg) to 10 ml with sufficient aq. pyridine to bring its final concn. to 40%. Heat on a water bath at 95° to 100° for 25 min., cool, add NaOH soln. (4%) (2 ml) and dilute to 25 ml with water. Measure the extinction within 30 min. The method can be used for the determination of chlorobenzene in the presence of benzene and toluene after nitration; an accuracy of about 5% can be attained.   P. S. STROSS

954. **Gas - liquid chromatography. Separation and micro-estimation of volatile aromatic amines.** A. T. James (Nat. Inst. Med. Res., Mill Hill, London). *Anal. Chem.*, 1956, **28** (10), 1564–1567.—The behaviour of a variety of substituted anilines has been studied on three chemically distinct gas - liquid chromatograms (paraffin wax or liquid paraffin, Lubrol MO, benzyldiphenyl). From the results it has been possible to formulate certain generalisations which should be useful in defining the conditions necessary for the separation of aromatic amines. These conditions should also apply to phenols and many other aromatic compounds. With these generalisations in mind it should be possible to identify almost any volatile aromatic amine by studying its behaviour on two or more types of column. Retention vol. relative to aniline at 137° are given for 61 aromatic amines on the three types of column used. K. A. PROCTOR

955. **The use of potassium periodate as a volumetric reagent [for hydrazine derivatives].** A. Berka and J. Zýka (Inst. Anal. Chem., Karlova Univ., Prague, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 314–316.—A 0·01 *M* solution of KIO_4 was used for direct potentiometric determination of some deriv. of hydrazine [phenylhydrazine (**I**), *iso*nicotinic acid hydrazide (**II**), semicarbazide (**III**), and thiosemicarbazide (**IV**)], cysteine (**V**), ascorbic acid (**VI**), thiourea (**VII**) and thiosinamine (**VIII**). **I**, **II**, **III** and **IV** were titrated in 9 *N* HCl, when KIO_4 was reduced to ICl (**I** must be dissolved in ethanol; **IV** must be first hydrolysed). In the titration of **V**, reduction of KIO_4 to iodine occurs, whilst in titrating **VI**, two inflection-points appear ($KIO_4 \rightarrow I_2 \rightarrow$ ICl). **VII** and **VIII** can be titrated in *N* HCl, when KIO_4 is reduced to KI. Detailed procedures for the titration of these compounds are given and the stoicheiometric relations are discussed. J. ZÝKA

956. **Detection with luminol in paper chromatography.** V. Moučka and P. Pařízek (Průmysl. škola chem., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 312–314.—3-Aminophthalhydrazide (luminol) when oxidised shows a characteristic blue luminescence, *e.g.*, when treated with H_2O_2 in alkaline medium in the presence of some organometallic complexes as catalysts. This phenomenon was used for the detection of luminol and related deriv. and also for detecting peroxidases or some cations (Fe^{3+}, Bi^{3+} and Hg^{2+}) after their previous chromatographic separation. A new method for the preparation and purification of luminol is described.   J. ZÝKA

957. **The distinction between flavone glucosides and flavones. VI. Analytical studies in the flavone field.** R. Neu (Firma Dr. Willmar Schwabe G.m.b.H., Karlsruhe, Germany). *Z. anal. Chem.*, 1956, **153** (2), 95–104.—The formation of complexes between metal ions and hydroxyflavones is reviewed. Chelates with Cd acetate are reported. Tables are given of the colours of the soln. or ppt. obtained by adding 0·1 *M* Cd acetate or 0·01 *M* Cu acetate in the presence or absence of acetic acid to 0·1% soln. of 21 various hydroxyflavones in methanol or dimethylformamide.   A. R. ROGERS

958. **High-frequency titration of organic bases.** C. Bertoglio Riolo and E. Marcon (Univ. of Pavia, Italy). *Ann. Chim., Roma*, 1956, **46** (7–8), 528–535.—A procedure for the titration of organic bases in glacial acetic acid solution with $HClO_4$, based on the measurement of dielectric constant at high frequency, is described. Titration curves so obtained, together with potentiometric titration curves, for phenazine, phenazine-N-oxide, benzo-α-phenazine and benzo-α-phenazine-N-oxide are shown.   L. A. O'NEILL

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959. Determination of aromatic hydrocarbons in reformed petrol by mass spectrometry. Shun Araki and Gen Nozawa (Inst. of Techno-anal. Chem., Faculty of Eng., Tokyo Univ., Hongo, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (6), 675–680.—The composition of aromatic hydrocarbons in reformed petrol containing only a negligible amount of olefins (directly distilled, platformed, hydroformed petrol, etc.) was studied with a mass spectrometer. The sample is fractionated into two groups of b.p. below and above 150°; the former consists mainly of hydrocarbons with 8 or fewer carbon atoms. By simplifying the simultaneous equations, the contents of xylenes (**I**), ethylbenzene (**II**) and styrene are determined from the peaks of m/e 106, 105, 104 and 103; those of **I**, **II**, toluene and n-propylbenzene are determined from the peaks m/e 106, 105, 92 and 91, whilst benzene is determined from the peak of m/e 78. For the elimination of the effect of the peaks due to isotopes, Washburn's formula (Mass Spectrometry, "Physical Methods in Chemical Analysis," Vol. 1, p. 587) was used. The results are in approximate agreement with those obtained by the H_2SO_4 absorption method or the aniline-point method.
K. Saito

960. Analysis of the hydrocarbons and impurities in liquid gas. N. Getoff and E. Sattler-Dornbacher (Inst. of Process Tech. and Technol. of Fuel, Vienna Tech. High School). *Prakt. Chem.*, 1956, **7** (6), 159–162.—A discussion is given of the methods used for estimating the hydrocarbons in commercial readily liquefiable mixtures (of lower paraffins which may contain olefins such as C_2H_4), such as natural gas, mixtures resulting from the high-pressure catalytic hydrogenation of coal, and mixtures made by oil cracking. The methods comprise separating the mixture into a number of narrow fractions and analysing the fractions for hydrocarbon content by elementary analysis (combustion for C) and by determination of physical or other properties (density, thermal conductivity, heat of combustion, etc.). When olefins (**I**) are present, separate portions of the fraction are subjected (a) to combustion analysis to find total hydrocarbons, and (b) to absorption to remove **I**, followed by combustion analysis of the residual saturated hydrocarbons (**II**), the content of **I** being given by the difference. Alternatively, the initial mixture may be subjected to absorption to remove **I**, and the residual **II** is subjected to fractionation with analysis of the fractions for **II**; the absorbed **I** can be desorbed, hydrogenated to **II**, and the product then fractionated and the fractions analysed for **II**; the value for **I** can thus be found indirectly. Methods discussed of separating the gas mixtures into narrow fractions include selective absorption and desorption (with active C), selective extraction (with liquid toluene), and low-temp. rectification (liquefying at very low temp., e.g., of liquid air, and rectifying with a low-temp. column). The uses of gas chromatography and of mass spectroscopy for separating the mixtures and analysis of the fractions are also discussed.
H. L. Whitehead

961. Anodic polarography of sulphur compounds in petroleum and its fractions. H. V. Drushel and J. F. Miller (Mellon Inst., Pittsburgh, Pa., U.S.A.). *Anal. Chim. Acta*, 1956, **15** (4), 389–393 (in English). —A semi-quant. anodic polarographic scanning procedure for detecting and estimating cyclic and non-cyclic aliphatic sulphides and alkyl aryl sulphides in crude oils and sulphur concentrates from petroleum is described. Use of a solvent-electrolyte, viz., methanol - nitrobenzene (3 + 7, by vol.) acidified to 0·2 M HCl, having min. sp. resistance and max. solvency, combined with pretreatment of the platinum-wire electrode, yields polarograms that are reproducible to within 1 or 2%. A silver - silver chloride reference electrode is used, cell resistances of 250 to 350 ohms being obtained. Pretreatment of the platinum electrode is effected by connecting the cell to a conductivity bridge (a.c. of 1000 c/s) for ≃ 15 min. at 25°. The method does not detect thiols, H_2S, S or di-tert.-butyl sulphide. Polarographic data for 16 typical organic sulphides are listed.
W. J. Baker

962. The polarographic determination of elemental sulphur in petroleum and its fractions. H. V. Drushel, J. F. Miller, W. Hubis and R. O. Clark (Mellon Inst., Pittsburgh, Pa., U.S.A.). *Anal. Chim. Acta*, 1956, **15** (4), 394–400 (in English).— An improved polarographic procedure for determining from ≃ 0·004 to 1·0% of S in crude petroleum is described. Acetic acid (0·09 M) and ammonium acetate (0·2 M) dissolved in a mixture of benzene and methanol (3:1, by vol.) is the solvent-electrolyte, methylene blue (10 mg per litre) is used as max. suppressor, whilst a silver - silver sulphide anode immersed in the electrolyte soln. replaces the bulky S.C.E. The reproducibility is within ≃1% and the accuracy is within 5%. The soln. should be initially degassed with N for ≃15 min., and afterwards blanketed with N while the polarogram, starting at −0·2 V vs. the silver - silver sulphide electrode, is recorded.
W. J. Baker

963. Method of indication of loss of naphthalene during its oxidation in reactors to phthalic anhydride. G. P. Korneĭchuk (L. V. Pisarzhevskiĭ Inst. of Phys. Chem., Acad. Sci., Ukr. SSR). *Zavod. Lab.*, 1956, **22** (8), 920.—A cotton-wool plug is placed in a tube fitted by means of a ground glass joint to the outlet tube of the reactor in which naphthalene is oxidised to phthalic anhydride over a vanadium catalyst. After 1 to 2 litres of gas has passed through the plug (3 to 5 min.), the plug is placed in a porcelain dish and treated with 0·3 to 0·5 ml of $CHCl_3$ and ≃ 0·2 g of dry $AlCl_3$ (Schwarz, *Ber.*, 1881, **14**, 1516). A green colour appears in the presence of ≮ 0·05 mg of naphthalene.
G. S. Smith

964. Detection of tar acids by paper chromatography. Kenji Negoro (Osaka Municipal Tech. Res. Inst., Kitaogimachi, Kita-ku, Osaka). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (5), 546–547. —Tar acids are converted into oxyacetic acids with chloroacetic acid and submitted to paper partition chromatography. For the developer, a mixture of n-butanol and aq. NH_3 soln. (sp.gr. 0·88) (4:1, by vol.) is the best. A neutral indicator, Na 2-(2:4-dinitrophenylazo)-1-naphthol-3:6-disulphonate was used for revealing the spots. The R_F values are as follows: 0·390 (phenoxyacetic acid), 0·482 (o-tolyloxyacetic acid), 0·515 (2:4-xylyloxyacetic acid), 0·555 (2:5-xylyloxyacetic acid), 0·479 (3:4-xylyloxyacetic acid), 0·519 (3:5-xylyloxyacetic acid), 0·425 (m-tolyloxyacetic acid), 0·437 (p-tolyloxyacetic acid), 0·674 (o-thymyloxyacetic acid) and 0·588 (p-thymyloxyacetic acid). By applying this method to the fractionated tar acids, most binary mixtures of the above-mentioned compounds can be separated.
K. Saito

965. Determination of small amounts of phenol in hydrocarbons. É. Kh. Rud' and S. Ya. Skochilova (Krasnoyarsk Synthetic Rubber Works). *Zavod. Lab.*, 1956, **22** (8), 919.—In the production of α-methylstyrene by oxidation of cumene followed by decomposition of the hydroperoxide and de-hydration of the dimethylphenylcarbinol, a side reaction of decomposition of the hydroperoxide can yield phenol, which acts as an inhibitor of the oxidation of cumene and interferes when α-methyl-styrene and butadiene are copolymerised. A method of determining phenol in α-methylstyrene is des-cribed. *Procedure*—The sample (100 to 120 g) is thrice extracted with vigorous shaking with 10 ml of 1% NaOH soln., and the combined extracts are diluted to 100 ml in a calibrated flask. An aliquot portion (50 ml) is mixed with 1 ml of a freshly prepared soln. of a diazonium salt, prepared by mixing in the cold equal vol. of 0·1% *p*-nitroaniline hydrochloride and 0·05% NaNO₂ soln. The colour intensity is measured against a standard in a Duboscq colorimeter. The method was tested over the range 0·001 to 0·01% with satisfactory results.
G. S. SMITH

966. Determination of carboxyl group in cellulosic materials by the "dynamic ion-exchange" method. H. Sobue and M. Okubo. *T.A.P.P.I.*, 1956, **39** (6), 415–417.—The pulp sample is charged into a reaction tube as a uniform cake, acidified with dil. HCl, washed with water and treated with an aqueous solution containing some cation. The cation ab-sorbed by the samples is determined from the change in cation concn. in the solution. Calcium is used as a cation, and EDTA for its determination. The method is claimed to be superior to the Ca acetate method.
S. V. SERGEANT

967. Rapid determination of long-chain alkyl sulphates attached to cellulose. Yuzi Takayama (Mitsubishi Rayon Co. Ltd., Kyobashi, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (7), 856–857.—Long-chain alkyl sulphates (**I**) extracted from cellulose with boiling water are mixed with rosaniline hydrochloride (**II**) and the product is extracted with a mixture of chloroform and ethyl acetate (1:1, by vol.) and determined colori-metrically at 530 mμ. **I** remains unhydrolysed on boiling in water for 20 min. The working curve is linear for 10 to 100 μg of lauryl sulphate per ml. No interference results from other constituents such as resin or colloidal sulphur. The error is ≃ ±2%. *Procedure*—The sample (0·1 to 1 g) is digested with 80 ml of boiling water for 5 min., cooled and made up to 100 ml (including the solid sample). A portion (1 to 4 ml) of the supernatant liquid is mixed with 1 ml of **II** soln. (13·5 mg of **II** is adjusted to pH 6·1 with 0·025 M phosphate buffer and made up to 100 ml; this is extracted with the solvent until the organic layer becomes colourless) and then shaken with the solvent (5 ml). A 3-ml portion of the solvent layer is made up to 10 ml with methanol and submitted to colorimetry.
K. SAITO

968. Identification of synthetic fibres by micro-fusion methods. D. G. Grabar and R. Haessly (Industrial Rayon Corp., Cleveland, Ohio, U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1586–1589.—Observa-tions are made on the hot-stage of a polarising microscope and reproducible melting-points are obtained by using a silicone oil as the mounting liquid to exclude air from the fibres during heating and to improve the microscopic image. Measure-ments of the melting-point and the eutectic temp.

of the fibre, with *p*-nitrophenol as a reference com-pound, are made and the characteristic behaviour of the fibres during heating and cooling are observed. Data are given for 13 synthetic fibres. G. P. COOK

969. Estimation of copper 8-quinolinate in mil-dewproofed fabrics. A. Rose, A. W. Hutchison, J. R. Hayes and I. R. Sharkey (Appl. Sci. Labora-tories Inc., State College, Pa., U.S.A.). *Amer. Dyest. Rep.*, 1956, **45** (12), 362–364.—Copper 8-quinolinate (**I**) in fabrics is estimated by boiling the sample in dil. H₂SO₄, adjusting the pH of the resulting solution to 6·0 and extracting **I** with chloroform, in which it is measured photometrically at 410 mμ.
S.C.I. ABSTR.

970. Determination of non-ionic surface-active agents by the use of the iodine - starch coloration. Yuzi Takayama (Mitsubishi Rayon Co. Ltd., Kyobashi, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (6), 661–663.—Lisk's iodine-starch method for the determination of polyoxy-ethylene stearate (**I**) (*Anal. Chem.*, 1951, **23**, 609) was extended to that of other polyoxyethylene non-ionic surface-active agents (> 40 p.p.m.) [polyoxy-ethylene-ureate (**II**), -phenylethers (**III**) or -aryl-ethers (**IV**)]. The use of the extinction coeff. at 372 mμ is better than that at 590 mμ. The measure-ment must be carried out at a definite temp. **II** and **III** are determined similarly to **I**. Since the reaction product of **III** with iodine - starch soln. has absorption max. at 280 and 223 mμ, it is also colorimetrically determined at 280 mμ. The colour obtained by the reaction of **IV** is so unstable that the extinction rapidly decreases with time. When the whole procedure is carried out in an ice bath, however, the error is <3%. The extinction de-creases in the presence of cationic and increases in the presence of anionic surface-active agents.
K. SAITO

971. Determination of anionic and cationic surface-active agents by the use of the iodine - starch coloration. Yuzi Takayama (Mitsubishi Rayon Co. Ltd., Kyobashi, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (6), 663–665.—The fact that anionic surface-active agents (**I**) such as the Na salt of sulphate esters of higher alcohols decrease the colour intensity produced by the action of iodine-starch reagent with a non-ionic surface-active agent [*e.g.*, polyoxyethylene stearate (**II**)] was applied to the analysis of **I**. The colour reaches max. intensity within 20 min. and remains unchanged for 40 min. The sample soln. containing 0·003 to 0·25% of **I** is mixed with starch soln. (80 ml of 1% acetic acid is brought to pH 5 with 4% aq. NaOH soln., made up to 100 ml, boiled with 0·8 g of starch, filtered and made up to 2 litres) (5 ml), 0·2% **II** (5 ml) and iodine soln. (0·05 g of iodine in 100 ml of 0·1% KI) and made up to 100 ml. The extinction is measured at 610 mμ after 40 min. A similar method is applied to the determination of cationic surface-active agents [cetylpyridinium chloride (**III**), stearyl-β-picolinium chloride (**IV**) and long-chain amino-alcohols (**V**)]. In this case the extinction of the iodine - starch soln. of **II** is increased. A sample soln. containing **III**, **IV** or **V** is similarly treated with the starch soln., **II** and the iodine soln. and the extinction is measured at 372 mμ. The colour is constant after 35 min. and remains unchanged for 30 min. **III** and **IV** have max. absorption at 260 and 265 mμ, respectively. They are also determined by the direct colorimetric method.
K. SAITO

972. Analysis of metal soaps. G. Marwedel (Schramberg, Germany). *Farbe u. Lack*, 1956, **62** (3), 92–96.—A solution (1 : 1) of the metal soap, *e.g.*, in white spirit or xylene, is dropped on to powdered oxalic acid and the mixture is warmed for a few minutes at 100°. Excess of oxalic acid and the separated soap acid is washed on to a filter with ethanol and toluene and, if required, the soap acid may be identified in the filtrate. For lead soaps, the metal may be determined directly by weighing the oxalate; Ba may be converted into $BaSO_4$ by treatment with H_2SO_4 on the filter; Ca, Ce, Co, Mg, Mn and Zn are converted into oxides or carbonates by ignition. The method is not suitable for soaps of Al, Fe or Sn. Separation of many mixed soaps, *e.g.*, those of Pb - Co, Pb - Mn, Pb - Zn, Ca - Co, Ca - Mn or Ca - Zn, is possible after conversion into oxalates. L. A. O'NEILL

973. Determination of cineole in volatile oils. F. H. L. van Os and J. H. Zwaving (Lab. voor Pharmacogn. der Rijksuniv. Groningen, Netherlands). *Pharm. Weekbl.*, 1956, **91** (22), 805–809.—Some aspects of Cocking's method are investigated with cineole and o-cresol of high purity. Methods are given for preparing (*a*) pure dry o-cresol with a freezing-point not below 30·5°, and (*b*) the cineole - o-cresol complex of freezing-point not below 55·2° (the B.P. value). *Procedure*—Weigh melted o-cresol (2·1 g) and thoroughly dried oil (3·0 g) in a test-tube; insert the thermometer and stir the mixture until it crystallises; note the highest reading of the thermometer. Warm the tube until its content is completely melted and insert it into the congealing-point apparatus and allow to cool slowly until the temp. has fallen to 3° below the initial value; then stir vigorously, rubbing the wall of the test-tube to encourage crystallisation and perhaps seed with a little of the pure cineole - o-cresol complex. The highest temp. reached is taken as the freezing-point. If this is below 27·4° add to the test-tube an equal wt. of the complex and repeat the test. If the freezing-point is not below 27·4° the cineole content is read from a table showing the percentage by wt. corresponding to the respective temp. from 24° to 55·2°. If the freezing-point has been determined with the addition of an equal wt. of complex, and the cineole content of the enrichment mixture read from the table is a %, then the cineole content (p) of the essential oil is $p = 2$ ($a - 50$) % w/w. P. HAAS

974. Quantitative analysis of turpentine oil by infra-red spectroscopy. I. Infra-red spectrophotometric determination of α-pinene and camphene. Tsuneichi Takeshita and Masatoshi Kitajima (Central Res. Lab., Japan Monopoly Co-operation, Shinagawa-ku, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (6), 645–648.—Infra-red spectra of α-pinene (**I**) and camphene (**II**) were examined and their use in the rapid determination of **I** (30 to 90%) and **II** was studied by the "baseline" method (Heigl *et al.*, *Anal. Chem.*, 1947, **19**, 293). The sample is placed in a cell of 0·05-mm thickness and the extinction measured at 772 cm^{-1} (for **I**) and 667 cm^{-1} (for **II**). The error of the result is <1% and <1·5%, respectively, whatever the other ingredients [β-pinene (**III**), limonene and tricyclene (**IV**)] in the sample.

    II. Infra-red spectrophotometric determination of β-pinene and tricyclene. Tsuneichi Takeshita and Masatoshi Kitajima. *Ibid.*, 1956, **59** (6), 648–649.—Similar experiments were carried out with **III** and **IV**. The intensity of absorption of

III at 855 cm^{-1} is independent of the amount of **I** in the sample; the determination of **III** (6 to 60%) is effected with an error of ±1%. **IV** has two characteristic absorption bands at 845 and 855 cm^{-1}; the latter is greatly affected in the presence of **II** by the broad band of C–H deformation bending vibration at 888 cm^{-1}. The error is ≃1·1% for **IV**. K. SAITO

975. Isopiestic method for determining molecular weights of polymers. R. L. Parrette (Aerojet-General Corp., Azusa, Calif., U.S.A.). *J. Polym. Sci.*, 1955, **15** (2), 447–458.—The isopiestic method is most suitable for polymers having mol. wt. between 1000 and 30,000. The usual isopiestic techniques have been modified to accelerate the attainment of isopiestic equilibrium and to eliminate water from the system. Results by this method are in adequate agreement with those by cryoscopy (for low mol. wt.) and osmometry (for high mol. wt.). B. J. WALBY

976. Observations on some modern methods for analysis of plastics. L. Fanica. *Chim. Anal.*, 1956, **38** (10), 353–361.—A review of existing methods of analysis as applied to plastics for the estimation of constituent elements and characteristic groups is given, with 28 references. D. G. FORBES

977. Ultra-violet spectrophotometric determination of quinol in methyl methacrylate. Tsugio Takeuchi, Motohisa Furusawa and Yuzi Takayama (Fac. of Eng., Yamanashi Univ., Motoyanagi-cho, Kofu). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (1), 118–121.—The absorption of methyl methacrylate (**I**) monomer in methanol is negligible at 295 mμ, where the max. absorption of quinol (**II**) is observed; the extinction coeff. of **II** is proportional to its concn. within the range 0·05 to 0·3 mg per 10 ml of methanol. When the **II** content in **I** is <0·001%, the extinction of the sample soln. is measured directly at 300 mμ, by placing a soln. of freshly prepared pure **I** in the compensating optical path. A linear working curve is thus obtained for 0·0005 to 0·002% of **II**. The presence of other ingredients such as methyl α-hydroxyisobutyrate, methacrylic acid (< 5 mg per 10 ml of methanol) or **I** polymer does not interfere with the estimation. K. SAITO

978. The micro-determination of chlorine in small samples of polymerised and copolymerised vinyl chloride. J. M. Bather (British Cotton Ind. Res. Ass., Shirley Inst., Didsbury, Manchester, England). *Analyst*, 1956, **81**, 536–540.—In the apparatus described and illustrated, the polymer is oxidised and the Cl evolved is absorbed and the resulting Cl^- are determined volumetrically. The apparatus consists of a right-angled bent reaction tube fitted to a horizontal absorption tube. The reaction tube is charged with the sample (≃ 1·5 mg), 75 mg of ceric ammonium sulphate and 0·5 ml of conc. H_2SO_4. The absorption tube contains 0·1 g of ferrous ammonium sulphate dissolved in 1 ml of water. The reaction tube is heated in a copper block to ≃350° for 50 sec. and the apparatus is then set aside for 2·5 hr., with the absorption tube horizontal. The contents of the absorption tube are rinsed out with water and the Cl^- are determined by the Volhard procedure. A blank soln. is treated similarly. A. O. JONES

979. Disc method for the determination of the melting-point of solid resins. S. Diamond (I.C.I. Ltd., Hexagon House, Manchester, England). *J. Oil Col. Chem. Ass.*, 1956, **39** (7), 472–477.—The method described is particularly suitable for the types of resin that froth on melting, *e.g.*, rosin-modified phenolics. The melting-point is that temp. at which a plug of the resin is forced out of an orifice under the pressure of 15 mm of Hg. The orifice ($\frac{7}{64}$ in. in diam. $\times$ $\frac{1}{8}$ in. deep) is formed in a circular plate ($\frac{47}{64}$ in. in diam. $\times$ $\frac{1}{8}$ in. deep) secured to the bottom of, and closing, a hollow cylinder ($1\frac{1}{2}$ in. deep $\times$ $\frac{1}{2}$ in. in internal diam.) filled with 15 g of Hg, and itself held in a Hg-filled heating block (level of the Hg in the bath is flush with the upper surface of the inner cylinder). After filling the orifice with the melted sample, and assembling and filling the apparatus, the temp. of the assembly is raised at the rate of 2° per min. from a temp. 40° below the expected m.p. At the m.p., the level of the Hg in the inner cylinder rises suddenly. Data on various resins are reported which indicate that melting-points by this method generally lie about 15° below those obtained by the Durrans or ring-and-ball methods.　　　　J. L. PROSSER

980. Determination of magnesium, total phosphorus and free phosphate in rubber latex. M. E. Tunnicliffe (Dunlop Rubber Co., Birmingham, England). *Trans. Inst. Rubber Ind.*, 1955, **31**, T141–T149.—A critical evaluation of the molybdenum-blue method for the determination of orthophosphate and Mg was made and the conditions for their determination in latex were established. Under the conditions of the test, $MgNH_4PO_4$ was completely pptd. A comparison of several reducing agents for the conversion of phosphate into molybdenum blue showed that $FeSO_4$ gives a stable reproducible colour which varies with the concn. of phosphate according to Beer's law. For the determination of total P, dry-ashing of the sample with $Mg(NO_3)_2$ is proposed. This should be followed by hydrolysis and determination of the phosphate in the ash as molybdenum blue, as for Mg. Free phosphate in latex serum is determined directly after coagulation and removal of interfering substances from the serum by cationic exchange. The procedures for the determinations of Mg, total P, and free phosphate are given.
　　　　　　　　　　　　　　CHEM. ABSTR.

981. The colorimetric determination of phenyl-2-naphthylamine in rubber. P. Menšík and D. Broulík (Research Inst. Synth. Rubber, Gottwaldov, Czechoslovakia). *Chem. Průmysl*, 1955, **5** (5), 212–213.—When treated with diazotised *p*-nitroaniline (I), phenyl-2-naphthylamine yields a red azo dye. A colorimetric determination of phenyl-2-naphthylamine in synthetic rubber, based on this reaction, has been developed. *Procedure*—Extract the sample for 3 hr. with acetone, add a 1% solution of I (1 ml) and read the extinction at 530 mμ. The method is accurate for 0·1 to 2·0% soln. of phenyl-2-naphthylamine.　　　　　J. ZÝKA

982. Ultra-violet determination of phenolic antioxidants in rubber. C. W. Wadelin (Goodyear Tire & Rubber Co., Akron, Ohio, U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1530–1531.—In extracts of synthetic rubber, phenols are the only substances that undergo shift in wavelength and increase in absorbance when the soln. are made basic. By choosing a wavelength where the antioxidant has a low absorbance in neutral soln. and a higher absorbance in 0·1 *N* KOH soln., the phenol content of rubber

may be determined accurately by measuring the increase in absorbance. Recoveries of added antioxidant to rubber were approximately 100%.
　　　　　　　　　　　　　　G. P. COOK

983. Differentiation of some natural tannin extracts by paper-chromatographic technique. L. W. Haddaway (U.S. Customs Lab., Baltimore, Md., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1624–1625.—The method is based on the selective absorption, on plain blotter squares, of various components of the tannins from a mixed solvent of diethyl ether, *n*-butanol and acetone.(2:3:3). The dried chromatogram is examined under u.v. light. The method is empirical, yet the distinctive coloured zones are adequate for the characterisation of many extracts. Data for 18 various tannin extracts are listed.
　　　　　　　　　　　　　　G. P. COOK

984. Determination of matter soluble in water [in leather]. Committee for Analysis of Vegetable-tanned Leather, Society of Leather Trades' Chemists ("Craigieburn," Duppas Hill Road, Croydon, England). *J. Soc. Leath. Tr. Chem.*, 1956, **40** (3), 89.—Degreased leather is exposed to air to evaporate all degreasing solvent, 500 ml of water at 25° is added, and the sample is rotated for 2 hr. at 55 to 60 r.p.m. The liquid is filtered and evaporated to dryness. The residue is dried to constant wt. at $\simeq 100°$. Details of apparatus are given.
　　　　　　　　　　　　　　B. R. HAZEL

985. Determination of sulphur dioxide in bleaching extracts [for leather]. Society of Leather Trades' Chemists ("Craigieburn," Duppas Hill Road, Croydon, England). *J. Soc. Leath. Tr. Chem.*, 1956, **40** (3), 89–90.—In this proposed official method, SO_2 is liberated from a solution of 1 to 2 g of bleaching extract by the addition of 20 ml of conc. HCl diluted with 500 ml of water. Carbon dioxide is passed into the flask containing the boiling soln. so that the liberated SO_2 is swept out into neutralised H_2O_2 contained in an attached Henville absorption tube. The H_2SO_4 formed is titrated against standard 0·1 *N* NaOH.　　　　B. R. HAZEL

986. Note on the silicon and aluminium content of chrome leather. C. A. Danon. *Rev. Tech. Ind. Cuir*, 1955, **47**, 168–170.—Errors in determining Al in chrome leather are chiefly due to the presence of Si, and incomplete pptn. of $Al(OH)_3$. A method is described in which Si is removed by boiling the ashed leather in HCl or HF and H_2SO_4, which gives a ppt. of SiO_2. Aluminium is determined in a solution of ashed leather dissolved in Na_2O_2 and acidified with HCl, by double pptn. with aq. NH_3. In the second pptn., hexamine is added.
　　　　　　　　　　　　　　B. R. HAZEL

987. Colorimetric determination of diphenylamine in LB.7T and LB.TU powders. J. Barlot. *Mem. Poudres*, 1954, **36**, 199–207.—Diphenylamine in powders containing dinitrotoluene and butyl phthalate can be determined colorimetrically by the reaction of diphenylamine sulphate with oxidants. Dissolve 2·5 g of powder in 100 ml of methanol and 50 ml of ethyl acetate. After solution is complete, add 120 ml of water and collect the ppt. on a sintered-glass disc. Heat the ppt. with 100 ml of methanol to 95° for 15 min. and then add water to 500 ml. Measure in a colorimeter, with a red filter. Use a 2-ml portion of sample with 3 ml of water to set the instrument at 100% transmittance. Make the determination by taking

2 ml of the sample soln. and adding 2 ml of $10 N$ H_2SO_4 and 1 ml of 10% $NaNO_3$ soln. Stir in a bath at 15°. When the soln. temp. reaches 20°, place the tube in the colorimeter. The colour value reaches a plateau, then decreases. Take the reading at the plateau and read off the diphenylamine in the sample with a calibration curve. The $NaNO_3$ used must contain about 0·4% of $NaNO_2$. The determination is as precise as the classical chemical determination. CHEM. ABSTR.

988. Determination of the oil content of putty. R. Paulson (166 Mill Lane, Codnor, Derbyshire, England). *J. Oil Col. Chem. Ass.*, 1956, **39** (7), 469–471.—A simple ashing procedure is described. The oil is destroyed by combustion at a temp. sufficient to convert the whiting into CaO, which is weighed as such. An alternative procedure is to ignite the sample in an atmosphere of CO_2 and weigh as $CaCO_3$. The sample (5 to 10 g) is weighed into a platinum crucible (silica crucibles disintegrate after 6 to 10 ashings; alumina crucibles are thermally unstable) and placed in a crucible furnace, preheated to 1000°, for 1 hr. After cooling for 30 min. over silica gel - NaOH in a vacuum desiccator, the residue is weighed. The oil content is given by $100 (x - y)/(100 - y)$, where x is the percentage weight loss of putty, and y is the percentage weight loss of the whiting under the same conditions of combustion, either the same whiting (probable total error 0·1%) or the same grade of whiting (probable total error 0·33%). J. L. PROSSER

See also Abstracts **799, 859, 871, 993, 1063, 1068, 1069, 1095.**

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE
Blood, Bile, Urine, etc.

989. The determination of magnesium and calcium in serum or plasma with ethylenediaminetetra-acetic acid (EDTA). A. A. Wilson (Boots Pure Drug Co. Ltd., Thurgarton, Notts., England). *J. Comp. Pathol.*, 1955, **65**, 285–290.—The Ca is pptd. as oxalate and the Mg from the supernatant liquid as $MgNH_4PO_4$ in a separate tube. The ppt. are dissolved in HNO_3. The Mg is titrated with EDTA (disodium salt) in the presence of hydroxylamine hydrochloride and Solochrome black WDFA; the contents of the tube are added to the soln. of Ca and the titration is completed. N. E.

990. Micro-determination of lead in biological fluids by oscillographic polarography. F. De Francesco. *Boll. Lab. Chim. Provenciali, Bologna*, 1955, **6**, 10–12.—Under the following operating conditions Pb gives a well-defined peak: bias 0·55 V, height of sweep 50 mV, frequency of sweep 25 peaks per min., dropping-mercury electrode with 1 drop every 6 min., in a $1·5 N$ NaOH soln. A special electrode assembly is used consisting of a capillary tube for the Hg and a capillary tube to the end of which is attached a platinum wire. Up to 15 mg of Pb per 100 ml can be determined. The organic matter in urine and blood is first eliminated by ashing, or electrolytically in HNO_3 soln. The Pb is extracted at pH 9 with a soln. of dithizone in CCl_4 or $CHCl_3$, the solvent is evaporated, 2 to 3 drops of conc. HNO_3 are added, the mixture is evaporated to dryness and ashed at a red heat, and the Pb is

taken up in 1 ml of $1·5 N$ NaOH. A standard curve is prepared from $0·01 M$ $Pb(NO_3)_2$ soln. CHEM. ABSTR.

991. The quantitative estimation of total bilirubin in urine. F. A. Gries and G. Gries (Med. Universitätsklinik, Marburg a. d. Lahn, Germany). *Klin. Wochschr.*, 1956, **34** (39–40), 1084–1088.— The method of With (*Hoppe-Seyl. Z.*, 1942, **275**, 166), with certain modifications, was found to give a reliable measure of the total bilirubin in urine. Mix 2 ml of urine with 1·5 ml of 11% $Na_2HPO_4.12H_2O$ soln. and 0·5 ml of 20% $CaCl_2.6H_2O$ soln. in a centrifuge tube and allow to stand, with frequent shaking, for 30 min. Centrifuge and wash the ppt. three times, or until the washings are colourless, with 0·5% $CaCl_2.6H_2O$ soln. Stir up the ppt. with 5 ml of 96% ethanol and add 1·5 ml of freshly prepared diazo reagent (Van den Bergh). After 30 min. add 2 ml of 25% HCl, transfer to a 10-ml calibrated flask and make up to the mark with 96% ethanol. After 30 min. measure the extinction in a 1-cm cell with filter S57 and calculate the bilirubin by means of a factor.
H. F. W. KIRKPATRICK

992. A new colour reaction for determining acetone, acetoacetic acid and β-hydroxybutyric acid in blood and urine. H. Schön and I. Lippach (Med. Universitätsklinik, Erlangen, Germany). *Klin. Wochschr.*, 1956, **34** (39–40), 1083–1084.—*Acetone and acetoacetic acid*—To 5 ml of fluoride blood add 5 ml of 20% trichloroacetic acid, shake well and filter. Place 4 ml of filtrate with 10 ml of saturated $NaHSO_4$ and a little water in a flask and steam-distil; collect 10 ml of distillate. *β-Hydroxybutyric acid*—To the residual fluid in the flask add 1 ml of 10% $K_2Cr_2O_7$ soln. and steam-distil a further 10 ml of distillate in about 8 min. *Colour test*—To 10 ml of distillate add 5 ml of 2:4-dinitrophenyl-hydrazine reagent (0·25 g in 100 ml of 30% $HClO_4$; stored in a brown bottle this soln. is stable for 3 to 4 weeks). Allow to stand at room temp. for 30 min. then extract the hydrazone with CCl_4 (20 ml, 10 ml), filtering the extracts through a dry filter-paper into a 50-ml calibrated flask. Add 0·2 ml of ethanolic KOH (0·1 N in absolute aldehyde-free ethanol), fill to the mark with absolute ethanol and shake until all turbidity disappears. Measure the colour and read the acetone concn. from a standard curve. The extinction is proportional to the concn. of acetone within the range 1 to 60 μg. For acetone bodies in urine, steam-distil 1 ml of urine with 10 ml of 10% H_2SO_4 and collect 10 ml of distillate. Oxidise the β-hydroxybutyric acid to acetone by adding 5 ml of 10% $K_2Cr_2O_7$ soln. to the residual liquid and steam-distil as before. Use 5 ml of distillate diluted with 5 ml of water for the colour test. H. F. W. KIRKPATRICK

993. Determination of trichloroethylene, trichloroacetic acid and trichloroethanol in urine. T. A. Seto and M. O. Schultze (Univ. Minn., St. Paul, Minn., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1625–1629.—The methods for the determination of trichloroethylene (**I**), trichloroacetic acid (**II**) and trichloroethanol (**III**) in urine are based on the Fujiwara pyridine - alkali reaction for the determination of certain chlorinated compounds. The crimson colour obtained on the addition of **I** and **II** to a pyridine - ethanolic KOH soln. is measured at 545 mμ for **I** and 530 mμ for **II**. Oxidation of **III** with a chromic oxide - nitric acid soln., is required before the colorimetric determination at

530 mμ. The methods were used in the study of the metabolism of trichloroethylene.   G. P. COOK

994. The estimation of 5-hydroxytryptamine in urine. F. Lembeck and K. Neuhold (Univ. Graz, Austria). *Naunyn-Schmiedebergs Arch. Exp. Path. Pharmak.*, 1955, **226**, 456–459.—Of two urine samples of 20 ml each, one is mixed with 1 μg per ml of hydroxytryptamine (**I**), both are adjusted to pH 3 to 4 with N HCl, evaporated under N at 100° to 5 ml, mixed with 2 g of NaCl and repeatedly extracted with 10 ml of acetone. The acetone extract is cooled for 6 hr. at $-10°$ to remove the NaCl, centrifuged, evaporated under N, and dissolved in 1 ml of H_2O. Paper chromatography is applied with *iso*propyl alcohol - H_2O (1:1) for 15 to 18 hr. whereby four parallel runs are applied —pure urine extract, urine extract plus 1 μg of **I**, 20 μg of **I** plus 0·05 ml of the first urine extract, and 20 μg of **I**. The R_F values are taken from the last two tests after applying diazo reagent. **I** and **I** - urine extracts have different R_F values. The corresponding spots on the first two runs are eluted and evaluated on the rat uterus. The morning urine of healthy persons contained 0·014 to 0·72 μg of **I**, that of a patient with carcinoid 3·12 to 3·45 μg.   CHEM. ABSTR.

995. Determination of creatine, creatinine, arginine, guanidinoacetic acid, guanidine and methylguanidine in biological fluids. J. F. Van Pilsum, R. P. Martin, E. Kito and J. Hess (Med. Sch., Univ. of Minnesota, Minneapolis, Minn., U.S.A.). *J. Biol. Chem.*, 1956, **222** (1), 225–236.—New methods are described for the determination of guanidino compounds. A modified Sakaguchi colour reaction for substituted guanidines is used in each procedure. Guanidinoacetic acid is determined by developing the colour reaction with a $Ba(OH)_2$ - $ZnSO_4$ protein-free filtrate. Arginine plus guanidinoacetic acid is measured by the colour reaction with a tungstic acid protein-free filtrate. Creatinine is degraded to methylguanidine for measurement, and creatine is converted into creatinine by heat and acid, and this is then degraded to methylguanidine. Methylguanidine and guanidine are determined by developing the colour reaction on a $Ba(OH)_2$ - $ZnSO_4$ protein-free filtrate (arginine free) which has been treated with a strong anion-exchange resin to remove guanidinoacetic acid. Any guanidine present must first be treated with dimethyl sulphate before the colour reaction is applied. Urinary creatinine values are, for all practical purposes, identical with those obtained with the alkaline picrate method.   J. N. ASHLEY

996. Location of pyrimidines on paper chromatograms. G. Harris and R. Parsons (Brewing Ind. Res. Foundation, Nutfield, Surrey, England). *Chem. & Ind.*, 1956, (44), 1312.—A chromatogram of pyrimidines is sprayed with 5% ethanolic phenol, dried at 100° and resprayed with commercial aq. NaClO. Cytosine, uracil and thymine give stable sky-blue colours. Adenine and hypoxanthine give similar colours but with lower sensitivity. The zones are easily distinguished from the differently coloured zones given by allantoin, urea, xanthine and guanine.   E. J. H. BIRCH

997. An improved method for the quantitative determination of amino acids by means of indanetrione hydrate. S. Jacobs (Nat. Inst. for Medical Res., Mill Hill, London). *Analyst*, 1956, **81**, 502–503.—In this improved method difficulties caused by variations in commercial samples of indanetrione hydrate are avoided by treating a soln. of the reagent in 2-methoxyethanol with a cation-exchange resin, recrystallisation being thus unnecessary. From a stock soln. thus purified the reagent is prepared by mixing 50 ml with 25 ml of water, 25 ml of a Na acetate - acetic acid buffer soln. (pH 5·5) and 0·08 g of $SnCl_2$. The amino acid in a Na citrate buffer soln. (2 ml) is mixed with an equal vol. of the reagent and the mixture is heated in a water bath at 100° for 30 min. The cooled mixture is diluted to 10 ml with aq. ethanol (1 + 1) and its extinction is measured spectrophotometrically.
                    A. O. JONES

998. Differences among some natural amino acids in their ability to reduce vanadic acid to vanadyl salts. J. García-Blanco and A. M. Pascual-Leone (Lab. fisiol. gen. C.-S.I.C., fac. med., Valencia, Spain). *Rev. Españ. Fisiol.*, 1955, **11**, 149–152.—Vanadic acid reagent (**I**) is prepared by adding 9 ml of conc. H_2SO_4 to a mixture of 10 mg of NH_4VO_3 and 1 ml of H_2O. A ppt. forms and redissolves in about 20 min. **I** is reduced by cystine, cysteine, methionine and tyrosine giving a blue colour; glycine, alanine, serine, valine, leucine, *iso*leucine, phenylalanine, lysine, arginine, aspartic and glutamic acids, and histidine fail to react; tryptophan gives a strong brown colour. Application to qual. analyses of amino-acid mixtures is discussed.
                    CHEM. ABSTR.

999. A spectrophotometric method for determination of cysteine and related compounds. Y. Avi-Dor and J. Mager (Israeli Inst. for Biol. Res., Ness Ziona, Israel). *J. Biol. Chem.*, 1956, **222** (1), 249–258.—When thiol compounds interact with fluoropyruvic acid a product is formed which has a characteristic u.v. spectrum with a max. at 265 to 275 mμ. With thiol compounds that have an unsubstituted amino group in the α- or β-position to the thiol group, the peak is shifted to 300 mμ and the mol. extinction is increased nearly 10 times. A spectrophotometric method based on these results is described for the determination of aminoethanethiol, cysteine, homocysteine, and related compounds. Reduced glutathione is determined by the same method after hydrolysis to cysteinylglycine. The method does not permit a distinction between cysteine, homocysteine and aminoethanethiol, but it may be useful in differentiating between cysteine and glutathione.   J. N. ASHLEY

1000. Use of dyes in electrophoretic analysis and immunoelectrophoresis in gelatinous medium. J. Uriel and P. Grabar (Inst. Pasteur, Service de Chemie, Microbienne). *Ann. Inst. Pasteur*, 1956, **90**, 427–440.—A technique is described whereby proteins, lipids, phospholipids and glycoproteins are subjected to electrophoresis on agar gel. The dried, transparent leaves of agar are treated with (*i*) azocarmine reagent (1 g of azocarmine, 450 ml of M acetic acid, 450 ml of ethanol and 100 ml of glycerol) to identify the proteins; (*ii*) a saturated solution of Sudan black in 60% ethanol to distinguish the lipids and lipoproteins; (*iii*) modified Schiff reagent and periodic acid to reveal the glycoproteins. The spots or lines of specific precipitates are either measured photometrically or photographed, as the colours are not permanent. By these methods, characterisations of either electrophoretic mobility plus chemical nature or electrophoretic mobility plus immunochemical specificity plus chemical nature are obtained. D. P. FELIX

1001. The differential periodic titration of proteins with N/150 potassium copper(III) tellurate. G. Beck (Med.-chem. Inst., Univ., Berne). *Mikrochim. Acta*, 1956, (4–6), 977–981.—Tervalent copper forms a stable tellurate complex which acts as an oxidising agent towards proteins, *e.g.*, cysteine is oxidised to cystine and then the peptide linkage is attacked. In the method described, 40 to 60 mg of protein is oxidised; the degradation curve proceeds in stages which may correspond to definite peptide linkages.
M. F. C. LADD

1002. Microchemical identification of radioactive and non-radioactive steroids. D. L. Berliner and H. A. Salhanick (Univ. of Utah College of Med., Salt Lake City, Utah, U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1608–1610.—The method is based on isotopic dilution and subsequent chromatography of the unknown substance and a derivative. About 20 μg of the postulated "authentic" steroid is added to about 50 counts per min. of the unknown radioactive substance; the specific activity is determined. The mixture is chromatographed and the position of the band determined by inspection with u.v. light. The area of the u.v. absorption and its radioactivity are measured. If the substances are identical they occupy the same region and the specific activity remains constant. As little as 0·01 μg of radioactive steroids and 20 μg of non-radioactive substances can be characterised.
G. P. COOK

1003. An isotope-derivative method for the detection and estimation of oestrogens. D. C. Leegwater (Utrecht Univ., Holland). *Nature*, 1956, **178**, 916–917.—The "pipsyl chloride" isotope dilution method (Keston *et al.*, *J. Amer. Chem. Soc.*, 1949, **71**, 249) for the determination of amino acids can be applied to the detection and determination of oestrogens in the blood of cows, etc. The sample ($\equiv 10$ μg of oestrogen) is esterified for $\simeq 15$ min. at 25° with 2 mg of pipsyl chloride (p-iodophenylsulphonyl chloride) labelled with ^{131}I in 0·4 ml of 75% acetone. The excess of reagent is hydrolysed by heating with 2% aq. borax soln. at 50° for 30 min., and the pipsyl deriv. of oestrone, oestradiol-17β and oestriol, plus some pipsyl chloride, are then extracted into benzene. The four compounds are separated completely by ascending paper chromatography, with S. & S. 2043 paper treated with 10% of liquid paraffin in ether, and a mixture of CCl_4 and liquid paraffin (7 : 2, by vol.) as solvent. The lower limit of detection is 0·3 μg of oestrogen. W. J. BAKER

1004. Chemical estimation of aldosterone in urine. A. J. Moolenaar (Dept. of Clin. Endocrinology, Univ. of Leyden, Holland). *Lancet*, 1956, **ii** (11), 556.—Half of a 24-hr. specimen of urine is hydrolysed for 24 hr. at pH 1 and room temp. and extracted with 5 portions of chloroform. The bulked extracts are washed with 5% Na_2CO_3 soln., then with water, and dried *in vacuo*. The residue is partitioned between toluene - ligroin - methanol - water (5 : 5 : 7 : 3) and the residue from the methanol layer is chromatographed on paper with toluene - octanol - methanol - water (98 : 2 : 50 : 50), when the aldosterone moves between cortisone and cortisol and separates well in 4 hr. The aldosterone fraction is eluted and chromatographed in a Bush system to separate an interfering chromogen and the 2 : 4-dinitrophenylhydrazone is then formed at 100°. The hydrazone ppt. is fixed with benzoic acid, washed 3 times with a saturated soln. of benzoic acid in dil. H_2SO_4, dissolved in 50% ethanol and

a pellet of solid NaOH is added. The colour is measured against an identically treated blank.
H. F. W. KIRKPATRICK

1005. The analysis of single cells. O. H. Lowry, N. R. Roberts and Mei-Ling W. Chang (Dept. of Pharmacol., Washington Univ. Sch. of Med., St. Louis, Mo., U.S.A.). *J. Biol. Chem.*, 1956, **222** (1), 97–107.—Methods are described for the determination of glutamic dehydrogenase, glutamic - aspartic transaminase and phosphoglucoisomerase in single large-cell bodies and other structures of similar size (0·005 to 0·03 μg dry wt.), together with a description of the difficulties to be avoided. For the determination of glutamic dehydrogenase a simple micro modification of a published method (Lowry *et al.*, *Anal. Abstr.*, 1956, **3**, 3730) is used. Glutamic - aspartic transaminase is determined by allowing the enzyme to act on a mixture of α-oxoglutarate and aspartate in the presence of DPNH and an excess of purified pig-heart malic dehydrogenase. The oxaloacetate formed is immediately reduced to malate and the DPN produced is measured fluorimetrically. Phosphoglucoisomerase is determined by a micro modification of a method to be published. A method for the determination of malic dehydrogenase is also described. This requires only 10^{-5} μg of dry brain, *i.e.*, one-two-thousandth the mass of a large single cell body. In this method the enzyme reduces oxaloacetate in the presence of DPNH, and the DPN formed is measured fluorimetrically. The standard error of this method is equivalent to about 15,000 mol. of the enzyme. The other methods give reproducible and accurate values with single anterior horn cell bodies, dorsal root ganglion cell bodies and neighbouring structures of the rabbit nervous system.
J. N. ASHLEY

Drugs

1006. Compleximetric titrations in pharmaceutical analysis. J. S. Faber (Rijks-Univ. Groningen, Holland). Thesis published by J. B. Wolters, Groningen, 1955, 250 pp.—This is a review with 188 references, but contains also much original work on the most suitable conditions for the compleximetric determination of Mg, Ca, Sr, Ba, Zn, Cd, Hg, Pb, Al and Bi in pharmaceutical products. The results are compared with those obtained by other methods.
N. E.

1007. The paper-chromatographic separation of alkaloids. J. Büchi and H. Schumacher (Pharmaz. Inst. E.T.H., Zurich). *Pharm. Acta Helv.*, 1956, **31** (9), 417–420.—Chromatograms are illustrated showing the separation of solanaceous alkaloids (**I**) [atropine, hyoscine and (+)- and (−)-hyoscyamine] and opium alkaloids (**II**) (morphine, codeine, cryptopine, thebaine, narceine, papaverine, narcotine, and a mixture of these) with good results. For **I**, the alkaloids (50 μg) are dissolved in methanol containing a slight excess of di-(p-toluoyl)-(−)-tartaric acid and put on Whatman No. 1 paper buffered at pH 5·8. The mobile phase was *iso*-butyl alcohol - toluene (1 : 1) saturated with H_2O ($\simeq 10$%). For **II**, the alkaloids (10 to 100 μg in 0·001 ml of $CHCl_3$ or methanol) are spotted on Whatman No. 1 paper buffered at pH 3·5. The mobile phase was as for **I**. In each case the descending method was used. After equilibration

for 48 hr. the chromatogram was run for about 3½ hr., giving a 30-cm descent. The paper was dried at room temp., the spots examined by u.v. light and then the paper was sprayed with Dragendorff's reagent.   D. P. FELIX

1008. The quantitative estimation of morphine in opium as its dinitrophenyl ether. A. B. Svendsen and E. D. Aarnes (Pharm. Inst. Univ., Oslo). *Sci. Pharm.*, 1955, **23** (1), 18–20.—A method is described for the quant. determination of morphine in opium by extracting the drug with methanol and ammonia followed by adsorption on to Al_2O_3 (Merck - Brockmann). The morphine - alumina mixture is eluted with chloroform - *iso*propyl alcohol and the eluate is treated with fluorodinitrobenzene in acetone for 4 hr. at room temp. The morphine dinitrophenyl ether is filtered off, washed, dried and weighed.
                    D. P. FELIX

1009. A method for the quantitative paper-chromatographic determination of the water-insoluble alkaloids of ergot. J. Kolšek (Inst. für org. Chem., Univ. Ljubljana, Yugoslavia). *Mikrochim. Acta*, 1956, (11), 1662–1671.—The individual water-insoluble alkaloids of ergot can be separated by ascending and descending paper chromatography with formamide as stationary phase and benzene or benzene - light petroleum (boiling range 40° to 75°) (6 : 4) as mobile phase. The R_F values given by Macek (*Pharmazie*, 1955, **10**, 422) are confirmed and extended; all the alkaloids of the ergotoxine group, ergotamine and ergosine, but not ergotaminine and ergosinine, can be separated from one another. The separated alkaloids can then be determined spectrophotometrically by the use of van Urk's reagent or *p*-dimethylaminobenzaldehyde. The method is suitable for the determination of the individual alkaloids in drugs, extracts, mixtures of isolated alkaloids and pharmaceutical preparations.
                    A. R. ROGERS

1010. Quantitative determination of individual water-insoluble alkaloids of Yugoslavian ergot. J. Kolšek (Inst. für org. Chem., Univ. Ljubljana, Yugoslavia). *Mikrochim. Acta*, 1956, (11), 1679–1684.—Five samples of ergot of Yugoslavian origin have been analysed for total water-insoluble alkaloids and by a paper-chromatographic procedure for the individual alkaloids (*cf. Anal. Abstr.*, 1957, **4**, 1009). The latter is the preferred method of evaluation. The lysergic acid content gives an indication of the proportion of dextrorotatory alkaloids present. Decomposition of the alkaloids to give free lysergic acid and rearrangement into the laevorotatory physiologically inactive alkaloids does not depend on the extraction process and is ascribed to faulty storage of the drug.   A. R. ROGERS

1011. The differential evaluation of aconite tubers and their preparations. V. K. Mital and H. Mühlemann (Pharmaz. Inst. Univ., Bern). *Pharm. Acta Helv.*, 1956, **31** (9), 420–427.—A quant. determination of aconite alkaloids by descending paper chromatography followed by spectrophotometry is described, by the use of Whatman No. 1 paper treated with 1% HCl, air dried, washed free of Cl⁻ with water, and again air dried. The alkaloidal extract was chromatographed with pure aconitine ($R_F = 0.62$) and benzoylaconine ($R_F = 0.47$) as markers. The mobile phase was amyl alcohol (freshly distilled), 25% formic acid and benzene (24 : 25 : 3). The alkaloids were located with Dragendorff's soln., and corresponding strips were

cut off the chromatogram of the unknown and eluted with 1% HCl. The eluate was examined spectrophotometrically at 233 to 234 mμ against an eluate from a blank control strip. It is also shown that conc. ethanol affects the alkaloids, whereas dilute ethanol as used for Tincture of Aconite (P. Helv.) does not.   D. P. FELIX

1012. A micro-scale spot-test for nicotine. P. Luís (Rosario 2021, Castelar, Buenos Aires, Argentina). *Analyst*, 1956, **81**, 548–551.—A test for nicotine, particularly suitable for forensic work, is described. An oxidising mixture of sodium bismuthate, mono- and di-sodium phosphates and glacial acetic acid is introduced into a capillary pipette with the test soln. and the mixture is moved away from the drawn-out end of the pipette, which is then sealed off in a micro burner. The mixture is tapped to the bottom of the tube and the tube is immersed in boiling water for 2 min. The liquid is centrifuged and the base of the tube containing the ppt. is cut away. The clear liquid is evaporated in a porcelain basin on the steam bath and, as evaporation proceeds, it assumes a blue or blue - green colour. The test will detect 0·075 μg of nicotine in 3 μl of soln. Many of the common alkaloids and organic bases do not interfere with the test; among those that do are members of the morphine group. Applications of the test to the detection of nicotine in urine and other materials are described.   A. O. JONES

1013. Determination of nicotine in tobacco. P. Luís and J. B. Niederl (Univ. de Buenos Aires, Fac. Ciencias Medicas, Argentina). *Mikrochim. Acta*, 1956, (4–6), 948–954 (in Spanish).—Luís's reaction for the detection of nicotine (*Rev. Asoc. Bioquim. Argentina*, 1953, **18**, 259) has been applied successfully to micro-scale work with Emich's capillary technique. The nicotine is oxidised with sodium bismuthate in soln. of controlled pH. The excess of oxidant is removed with oxalic acid and the colour reaction with conc. H_2SO_4 is carried out on the residue. The sensitivity is 0·03 μg in 3 μl.
                    M. F. C. LADD

1014. The determination of bacitracin and neomycin in mixtures. J. Lingnau and G. Machek (Forschlab. Biochem. Ges. m.b.H. Kundl, Tirol). *Sci. Pharm.*, 1955, **23** (4), 234–240.—A method is described for the separation and determination of bacitracin and neomycin in mixtures by means of the solubility difference in ethanol. After centrifuging an ethanolic extract the sol. bacitracin can be removed and determined microbiologically with *Micrococcus flavus*. The insoluble neomycin sulphate is dissolved in phosphate buffer at pH 8·0 and determined with *Bacillus subtilis*. Modifications are described to make the method applicable to tablets, powders, dry mixtures and ointments.
                    D. P. FELIX

1015. Microbiological assays of novobiocin in pharmaceuticals and biological fluids. A. Kirshbaum, J. Kramer, B. Arret and B. M. Wintermere (Food and Drug Admin., Washington, D.C., U.S.A.). *Antibiot. & Chemother.*, 1956, **6** (8), 504–510.—Details are given of a plate assay procedure for novobiocin (**I**) in pharmaceutical preparations with *Micrococcus pyogenes* var. *albus* (ATCC12228) as test organism. The method is suitable for concn. of **I** about 1·0 μg per ml (standard-curve range, 0·6 to 1·5 μg per ml). Neither dihydrostreptomycin (100 μg per ml) nor tetracycline antibiotics (200 μg per ml) interfere. A turbidimetric method with *M. pyogenes* var. *aureus* (PCI 1209 RD) and suitable

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for concn. between 0·3 and 0·8 μg per ml is also described. Serum concn. of **I** in the range 1·5 to 80 μg per ml may be determined by a plate assay with *Sarcina lutea* (PCI 1001, ATCC 9341) and, with the modifications given, the sensitivity may be increased to detect 0·5 μg per ml without preliminary extraction. In urine, a concn. of 1 μg per ml of **I** may be determined by a plate assay with the same test organism.　　W. H. C. SHAW

1016. Determination of some pharmaceuticals by bromination in anhydrous acetic acid. B. Miszczuk-Łucka and H. Taborska (Zakład Chem. Inst. Lekōw, Poland). *Przem. Chem.*, 1955, **11**, 706–710.—In the method described, a soln. of Br in pure anhyd. acetic acid is used. It was found that the purity of glacial acetic acid (**I**) used seriously affected the stability of the Br soln., and that the best method for purification and dehydration of commercial **I** (96%) was by freezing, followed by distillation over CrO₃ and re-freezing. *Procedure*—The sample (0·05 to 0·2 g) was weighed into a conical flask and dissolved in 10 to 30 ml of anhyd. **I** (samples containing more than 1% of moisture were previously dried). The calculated amount of the Br soln., with the required excess, was then added from a burette, with constant mixing. The flask was stoppered and set aside in the dark at 18° to 20°. The time of reaction (10 to 60 min.) depended on the substance tested. The mixture was then treated with KI soln. (10%) (10 ml) and the liberated I was titrated at once with 0·1 *N* thiosulphate, with starch as indicator. Details of optimum conditions are given for salicylamide, phenazone, thymol, procaine, dihydrocodeine tartrate, dihydrodihydroxycodeinone hydrochloride, diamorphine hydrochloride, sulphanilamide, sulphathiazole, sulphadimidine, sulphaguanidine, vanillin *iso*nicotinohydrazone, isoniazid and *p*-hydroxyacetophenone. Phenacetin could not be determined by this method.

K. F. SPOREK

1017. Colorimetric determination of the individual components of acetylsalicylic acid, acetophenetidine [phenacetin] and caffeine mixtures. R. E. Pankratz and F. J. Bandelin (Control Lab., Flint, Eaton & Co., Decatur, Ill., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1956, **45** (6), 364–366.—The individual colorimetric determinations of acetylsalicylic acid, phenacetin and caffeine are described. *Procedure for acetylsalicylic acid*—Crushed tablets (equiv. to 1 g of acid) are dissolved in 25 ml of 5% NaHCO₃ soln., and extracted with CHCl₃ (3 × 25 ml), each extract being washed with 15 ml of NaHCO₃ soln. The combined aq. extracts are warmed with 50 ml of alcoholic KOH for 2 hr., then cooled, and 5 ml of conc. HCl is added and the soln. is diluted to 250 ml. A 20-ml aliquot is further diluted to 100 ml, and to 5 ml of this soln. is added 5 ml of Fe(NO₃)₃ soln. [1 g of Fe(NO₃)₃.9H₂O in 100 ml of HNO₃ (1 : 100)] and diluted to 100 ml, and the extinction is determined at 525 mμ. *Phenacetin*—A powdered sample (containing 0·1 g) is treated with 20 ml of 10% H₂SO₄ on a water bath for 3 hr., then cooled, and a 10-ml portion is diluted to 200 ml with water. A 10-ml aliquot is treated with 15 ml of water, 1 ml of dil. HCl and 1 ml of 1% NaNO₂ soln. and set aside for 3 min. Buffer soln. (5 g of NaHCO₃ and 1 g of NaOH in 100 ml of water) (20 ml), followed by 1 ml of 2-naphthol soln. (1% in acetone), is added. After 15 min., 40 ml of acetone is added and, after further dilution, the extinction is measured at 470 mμ. *Caffeine*—A sample containing 100 mg is dissolved in 100 ml of water and

filtered; a 5-ml aliquot is diluted to 25 ml, and 1 ml of dil. HCl and 1 ml of 10% molybdophosphoric acid are added. The mixture is heated on a water bath for 15 min., then chilled in ice, and centrifuged. The ppt. is washed with dil. HCl, dissolved in 25 ml of acetone, and the extinction is measured at 440 mμ. Comparisons with standard curves are required. The results with compounded tablets show recoveries of acetylsalicylic acid of 100·6, phenacetin 99·2 and caffeine 100·2% of theoretical.　　G. R. WHALLEY

1018. Detection and identification of clinically important barbiturates. L. Levi and C. E. Hubley (Dept. of Nat. Health and Welfare, Ottawa, Ont., Canada). *Anal. Chem.*, 1956, **28** (10), 1591–1605.—The barbiturates can be identified by inspection of the i.r. absorption spectra of their copper - pyridine complexes as well as the i.r. absorption spectra of the free barbituric acids recovered from the complexes. The compounds show unique features throughout the 4000 to 650-cm⁻¹ region, which afford a high degree of specificity for detecting and characterising these drugs. The mechanism of the reaction of barbiturates with the copper - pyridine soln. was extensively studied and the composition of the dark-purple complex was investigated. The reaction becomes more sensitive as the CuSO₄ to pyridine ratio in the reagent is increased or the pyridine to water ratio of the system is decreased. Absorption spectra of 12 barbituric acids and their complexes are illustrated.　　G. P. COOK

1019. Comparative studies of methods for assay of benzocaine. C. E. Fischbach E., C. López Pérez and J. Mata Vázquez (Invest. Dept., Customs Lab., Ministerio de Hacienda). *Acta Chem. Venezuela*, 1956, **7** (1), 14–16.—Descriptions are given of three methods of determining the purity of benzocaine— (*i*) by diazotisation of 0·5 g in HCl (5 ml) and water (50 ml), after cooling to 15° and adding 25 g of broken ice, with 0·1 *N* NaNO₂ to a blue end-point with starch - iodide paper; the colour is permanent after 1 min.; (*ii*) bromination of 40 mg in 60 to 80% acetic acid (10 ml) and water (90 ml) by addition of 0·1 *N* bromide - bromate solution (20 ml), followed by acidification with HCl (5 ml) and titration of the excess of Br with 0·1 *N* Na₂S₂O₃ after addition of 1 g of KI in 5 ml of water; and (*iii*) titration of 0·3 g in glacial acetic acid (20 ml) with 0·1 *N* HClO₄ in glacial acetic acid (made by dissolving 70% aq. HClO₄ in acetic acid and adding acetic anhydride to take up the water), with 1% methyl violet in acetic acid as indicator. The last method is suitable for the determination of benzocaine in ointments with non-fatty bases. The methods are of similar precision but differ in rapidity and simplicity. Bromination gives satisfactory results and is simple and rapid. Titration in acetic acid is the most recommended method, both for pure benzocaine and for pharmaceutical preparations, provided that these contain no basic substances or if the benzocaine can be separated quantitatively.

D. LEIGHTON

1020. The determination of benzocaine in galenical preparations. G. Machek (Forschlab. Biochem. Ges. m.b.H., Kundl, Tirol). *Sci. Pharm.*, 1956, **24** (3), 151–154.—A method is described for the quant. determination of benzocaine in preparations, *e.g.*, tablets, ampoules, suppositories. A solution containing approx. 3 μg of benzocaine per ml dissolved in 0·1 *N* HCl is diazotised with 0·1% NaNO₂ followed by treatment with ammonium sulphamate at 10° and coupled with N-1-naphthylethylene-

diamine hydrochloride (Bratton - Marshall, *J. Biol. Chem.*, 1939, **128**, 537). The red - violet colour quickly reaches a maximum and is stable for at least 3 hr. The colour intensity is read in a colorimeter at 525 mμ, and the results compared with a standard graph.　　　　　　D. P. FELIX

1021. Determination of atophan [cinchophen]. M. Vietti-Michelina and R. Pilleri (Turin Univ., Italy). *Anal. Chim. Acta*, 1956, **15** (4), 324–325.—Cinchophen can be determined accurately in the presence of salicylic and acetylsalicylic acids if the three acids are present as their ammonium salts. To 10 ml of ammoniacal soln., at 100°, is added, dropwise, 20 ml of 2% aq. ammonium molybdate in the presence of 10 ml of glacial acetic acid. After remaining for 4 hr. on the steam bath, the ppt. is filtered off, washed, dried (105°) and weighed.
　　　　　　W. J. BAKER

1022. A combined oxidation- and distillation-apparatus for toxicological analysis. W. Croon (Staatl. Inst. für Hygiene und Infektionskrankheiten, Saarbrücken). *Z. anal. Chem.*, 1956, **153** (2), 105–107.—A large three-way tap allows connection of a long-necked flask either to a reflux condenser or by way of a sloping condenser to a receiver. Large samples (*e.g.*, 50 g) can be oxidised rapidly by alternately refluxing with oxidising agent and removing the reaction products by distillation.
　　　　　　A. R. ROGERS

See also Abstracts 973, 974, 994, 1039, 1062.

Food

1023. Potentiometric titrations in food analysis. K. Woidich, L. Schmid and H. Gnauer (II Chem. Univ. Lab., Vienna). *Z. LebensmittUntersuch.*, 1956, **104** (2), 97–104.—The suitability of electrometric titration based on the analytical process of Cruse (*Angew. Chem.*, 1953, **65**, 232) and the graphical method of Tubbs (*Anal. Abstr.*, 1955, **2**, 267) for the determination of the equivalence point and pH range from the titration curve, is discussed in a series of test analyses. Especially useful results were obtained for the determination of acid in jam, flour, sugar and vanillin. The tests were made with the Beckman automatic titrator model K, glass electrode 4990–83 and the calomel electrode 1170–71, and standard aq. buffer soln. of pH 7·0, 3·0 and 10·0. Adaptations of the method to improve accuracy and save time are described.
　　　　　　S.C.I. ABSTR.

1024. Semi-micro compleximetric estimation of phosphoric acid in food samples. R. Brochon and H. Eschmann (Lab. Cantonal, Berne). *Mitt. Lebensmitt. Hyg., Bern*, 1956, **47**, 155–182.—A review of the commoner methods for the estimation of H_3PO_4 is given and a method for the rapid semi-micro determination of PO_4^{3-} is proposed which depends on the pptn. of P in the form of $MgNH_4PO_4$. The main innovation is the use of ethanolamine to render the soln. alkaline. The method is applicable to a wide range of foodstuffs without variation of technique, and is reliable in the presence of common ions. By the addition of known amounts of phosphate to the sample, traces of $\simeq 1$ mg can be accurately determined. *Procedure.*—Mix Mg acetate soln. (0·1 M) (10 ml) with $\not> 5$ g of sample containing $\not> 50$ mg of PO_4^{3-} (or the equiv. quantity of a liquid sample) in a platinum dish, evaporate to dryness on a water bath and dry if necessary at 105°, or at 150° for sugary samples. Ignite and

triturate the cooled ash with H_2O (10 to 15 ml), followed by N H_2SO_4 (20 ml), filter and wash the residue with boiling water (3 × 20 ml). Ignite the filter and residue, dissolve the ash in N H_2SO_4 (10 ml), and filter as before. Evaporate the liquid to a vol. of 5 to 10 ml under a 50-cm air condenser in order to oxidise meta- and pyro-phosphates. Cool the liquid and dilute to 50 ml. Make a preliminary titration of 5 ml of the soln. with EDTA (0·1 M), with Eriochrome black T as indicator, to determine the equiv. of interfering ions. Mix 20 ml of the sample soln. with lactic acid (0·5 ml), EDTA (0·1 M) (equiv. to the interfering ions, with 0·5 ml in excess) and methyl red indicator (2 drops). Stir the mixture magnetically and neutralise with 2 N aq. NH_3. Titrate with a soln. of ethanolamine (5%) to phenolphthalein. Filter off the ppt. and wash it with ammoniacal ammonium chloride soln. (N aq. NH_3 - 0·1 N NH_4Cl). Dissolve the ppt. in N HCl (5 ml), wash the filter with boiling 0·2 N HCl and finally with boiling water (2 × 20 ml) just acidified with HCl. To this soln. add 0·01 M EDTA (25 ml), 2 N aq. NH_3 (20 ml), and methyl red indicator (6 drops). Titrate the excess of EDTA with 0·01 M $MgSO_4$, with Eriochrome black T as indicator.　　　　　　D. G. FORBES

1025. Paper chromatography of sugars in foods. E. Becker (Wissenschaft. Lab. Dtsch. Arkady - G.m.b.H., Hannover, Germany). *Z. Lebensmitt-Untersuch.*, 1956, **104** (2), 122–126.—A method for sugar determination with *n*-butanol - pyridine - water and development of colour of (*a*) reducing sugar with phthalic acid - aniline, and (*b*) non-reducing sugar with 1 : 3-dihydroxynaphthalene - trichloroacetic acid is described. The chromatographed sugars are taken up in water. The method is semi-quant., and with practice gives results of an accuracy within $\pm 10\%$.　　　　　　S.C.I. ABSTR.

1026. Influence of hydrochloric acid on hydrolytic decomposition of starch and starchy raw materials in connection with the quantitative determination of starch. M. Krajčinović and J. Djukić-Jovanović (Tehn. Fac., Univ. Zagreb, Yugoslavia). *Z. Lebensmitt-Untersuch.*, 1956, **103** (5), 350–355.—Analytical procedures based on the hydrolysis by HCl of maize, maize starch, or distiller's wash, followed by the determination of reducing power of the hydrolysate, fail to give results for starch in accordance with results obtained by the standard method of Lintner or of Ewers. This is explained by the presence in the HCl hydrolysate of unhydrolysed dextrin and hydrolytic products of cellulose and pentosans.　　　　　　P. S. ARUP

1027. The use of ion exchangers for determining the grade of flour. J. Pomeranz and C. Lindner (Min. Trade Ind., Haifa, Israel). *Anal. Chim. Acta*, 1956, **15** (4), 330–334 (in English).—The water-sol. electrolyte content (E) of flour can be determined by passing a filtered suspension (at 25° $\pm$ 2°) of the sample (25 g) through a 25-cm column of Amberlite JR-120 (H form) and titrating the liberated acid in the eluate. A separate titration of any acid originally present in addition to the mineral salts should also be made. The method, which is valid in the presence of added $CaCO_3$, can replace the usual ash test for evaluating the extraction rate of flour. A linear equation enables the "original" ash content to be calculated approx. from E, provided that the sample is not pure bran. A modified Feigl spot-test for determining the amount of added $CaCO_3$ to within 0·1% is described. W. J. BAKER

1028. Determination of fat in meat and meat products by Gerber procedure. M. S. Pohja, S. E. Komulainen and F. P. Niinivaara (Osuusteurastamojen Tutkimuslaitos, Hämeenlinna, Finland). *Z. LebensmittUntersuch.*, 1956, **103** (5), 333–341.— The van Gulik method for determining fat in cheese can be used with slight modifications for the determination of fat in meat and meat products. The van Gulik procedure works well with a sample wt. of 2·5 g in butyrometer tubes designed to give direct readings for 3 g of cheese. The time for digestion of the sample with H_2SO_4 (sp.gr. 1·53) is 30 to 45 min. The results are vitiated by carbonisation only in the presence of entrails and digestive organs. Single results show max. deviations of $\pm 0·4 \%$ from results obtained by the Schmid - Bondzynski - Ratzlaff method. Average results are in good agreement with those obtained by the latter method, and are $\simeq 1\%$ higher than those obtained by the Soxhlet extraction method.   P. S. ARUP

1029. Causes for variations in xanthine dehydrogenase activity in cows' milk from different districts. F. Kiermeier and K. Vogt (Süddeutsch. Versuchs- u. Forsch. Anstalt f. Milchwirtsch., Weihenstephan, Germany). *Z. LebensmittUntersuch.*, 1956, **103** (5), 355–361.—Considerable regional variations occur in the enzymic activity of milk, measured by its capacity for reducing methylene blue in the presence of formaldehyde (Schardinger test). A high order of positive correlation is found between this enzymic activity and the alkalinity of the soil of the district.   P. S. ARUP

1030. Determination of free and combined formaldehyde using modified chromotropic acid procedure. Application to determination of piperine content of pepper. L. A. Lee (Eastern Utilisation Res. Branch, USDA, Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1621–1623.—The method is based on the quant. determination of formaldehyde liberated from the methylenedioxy groups in piperine by acid hydrolysis. The formaldehyde is measured by the chromotropic acid colorimetric procedure. The method was applied successfully to ethanolic extracts of pepper, a standard deviation of $\pm 0·43\%$ being obtained. Recoveries $>99·4\%$ with a standard deviation of $\pm 0·52\%$ were obtained from a pure piperine sample.   G. P. COOK

1031. Investigation of emulsifying agents in beverages [II]. G. Janssens and Y. Lutz (Lab. Central, Min. de l'Agric., France). *Ann. Falsif.*, 1956, **49**, 348–356.—Methods of distinguishing cellulosic ethers and sorbitan esters in the presence of each other are discussed, and a number of emulsifying agents, which may be used (though forbidden in France) are listed. Reactions by which methylcelluloses and cellulose ethers of polyalcohols can be characterised are tabulated, and some of their uses are given. The characterisation of gum benzoin (used to give a cloudiness in the presence of emulsifying agents) by its colour with copper acetate is discussed.   E. J. H. BIRCH

1032. Titrimetric determination of carbon dioxide in beer. G. Nowak. *Brauwelt, B,* 1956, **96** (51–52), 865–866.—A measured quantity of beer is treated with an excess of NaOH soln. of known concn. and the mixture (10 ml) is titrated with 0·2 N acid to pH 8·3 (phenolphthalein), the CO_2 being converted into $NaHCO_3$ (*a*). A second sample of beer is boiled, then cooled, and a 10-ml portion is titrated with 0·2 N NaOH to pH 8·3

(phenolphthalein). This value gives the other acids present (*b*). If the amount of NaOH soln. used at first $\equiv x$ ml of 0·2 N NaOH, then the CO_2 of the beer has combined with $x - (a + b)$ ml of 0·2 N NaOH, and is thus the measure of CO_2, grams per litre, of beer.   S.C.I. ABSTR.

1033. Routine determination of sulphate in wines. J. Schneyder (Landwirtschaft. Bundesversuchsanstalt, Vienna, Austria). *Mitt. Wein-u. Obstbau, Wien, A,* 1956, **6** (4), 155–157.—This rapid method gives results in good agreement with gravimetric results. *Procedure*—The sample (25 ml), contained in a centrifuge tube, is mixed successively with ethanol (2 to 3 ml), conc. HNO_3 (0·6 ml), and aq. 25% $Pb(NO_3)_2$ soln. containing 1·5% (v/v) of conc. HNO_3. The $PbSO_4$, separated by centrifuging, is washed with dil. ethanolic HNO_3, and transferred to a flask in which it is dissolved in a known excess of 0·1 M EDTA (disodium salt) soln. After the addition of a buffer soln. (2 ml) containing NH_4Cl and aq. NH_3, the excess of EDTA is back-titrated with 0·1 M $ZnCl_2$ soln., with Eriochrome black T as indicator.   P. S. ARUP

1034. Determination of dry matter in emulsion-liqueurs by means of infra-red radiation. J. Kottász (State Inst. for Chem. and Food Examn., Budapest, Hungary). *Z. LebensmittUntersuch.*, 1956, **103** (5), 386–387.—Drying by means of i.r. radiation affords a convenient and accurate procedure for the determination of dry solids in "egg-brandy," ice-cream and similar products.   P. S. ARUP

1035. Extraction and identification of synthetic dyestuffs incorporated in food. I. Saenz-Lascano-Ruiz (Lab. Municipal, Paris). *Ann. Falsif.*, 1956, **49**, 315–332.—Methods of extraction of dyestuffs from food samples are given for four classes— beverages where the dye is in solution, essentially glucosic materials (sweets and pastries), protein substances and fatty substances. Separation of the dyestuffs thus extracted is effected on an alumina column, and they are classified by the methods of Rota and Green (the latter method with some modification, owing to the small quantities involved). The dyestuffs are identified by paper chromatography and a list of R_F values for different eluents on Arches 302 paper is given for dyestuffs permitted in France. The characteristic reactions of permitted natural dyestuffs are also reviewed.   E. J. H. BIRCH

1036. The determination of iodine values by the Kaufmann method. H. Steindel (Zakładu Anal. i Technol. Leku Roślinnego, Poland). *Biul. Nauk.*, 1956, **2** (3), 160–163.—A comparison was made between the Hübl method, with iodine, and the Kaufmann method, with bromine, for the determination of the iodine value of unsaturated fats. The Kaufmann method, in which a stable reagent is used, was found to give reproducible results and to require a much shorter time for completion of the reaction. *Recommended procedure*—Weigh into a conical flask with ground-glass neck an amount of the fat sample according to its iodine value, as follows—iodine value 20, 1 g; 20 to 60, 0·5 g; 60 to 120, 0·25 g; over 120, 0·1 to 0·15 g. Dissolve the sample in 10 ml of $CHCl_3$ and add 25 ml of the bromine reagent (dissolve 12 to 15 parts of NaBr, previously dried at 130°, in 100 parts of methanol, decant the clear solution and add 5·2 ml of Br per litre). Close the flask with a glass stopper and set aside in the dark, for 30 min. for fats with low

iodine values and for 2 hr. for those with high values. Then add 10% KI (15 ml) and titrate with 0·1 N Na₂S₂O₃. Carry out a blank determination in the same way. For fats with high iodine values the reaction can be speeded up by immersing the flask in a water bath at 40° to 50° for 30 min.

K. F. SPOREK

1037. Determination of linolenic acid from hexabromide value. K. D. Pathak and J. S. Aggarwal (Nat. Chem. Lab., Poona, India). *J. Sci. Ind. Res., B, India,* 1956, **15** (5), 247–250.—Linseed-oil fatty acids (1·5 g, or $\simeq$ 1 g for linolenic acid concentrate) are placed in a weighed 50-ml centrifuge tube. The glass rod used in the determination is weighed separately. The acids are dissolved in ether (30 ml) and cooled to a temp. of 0° to 5° in an ice - salt bath. Bromine is added dropwise from a pipette, with stirring after each addition, until the soln. becomes permanently reddish-orange. After being set aside at 0° to 5° for at least 4 hr. the tube is centrifuged and then allowed to stand in the ice bath for 3 to 5 min. The supernatant soln. is poured off, the residual bromides are washed with 30-ml portions of ether reagent with efficient stirring, centrifuged, cooled and decanted each time. The solvent is removed by keeping the tube at about 60° to constant weight. The amount of acids taken for the determination is so adjusted that the final wt. of the ppt. is in the range 0·5 to 0·6 g. The procedure is satisfactory for amounts of fatty acid mixtures $\not>$ 6 g. The method gives results that are comparable to those obtained by the u.v. spectroscopic method.   I. JONES

1038. Urea adducts of fatty acids. X. Estimation of the adulteration of mustard oil with linseed and groundnut oils. T. N. Mehta, B. Y. Rao, S. M. Abhyankar and B. H. Alase (Laxminarayan Inst. Technol., Nagpur Univ., Nagpur M.P.). *J. Indian Chem. Soc., Ind. Ed.,* 1956, **19** (1), 39–47.—The urea-adduct separation technique is used to estimate the adulteration in mustard oil with linseed and groundnut oils by concentrating the characteristic fatty acid, erucic acid. The percentage yield of C₂₂ unsaturated acid in the adduct-forming fraction is calculated from its iodine and neutralisation values to determine the percentages of mixed oils in mustard oil.   I. JONES

1039. Colorimetric determination of folic acid in presence of iron salts. S. K. Ganguly and Hrishikesh Bhattacharya (Bengal Immunity Res. Inst., Calcutta, India). *Indian J. Pharm.,* 1956, **18** (9), 361–364.—Folic acid in liver extract and soln. of vitamin B containing FeSO₄, ferric ammonium citrate or ferrous gluconate can be determined by the usual colorimetric method with pteroylglutamic acid (Hutchings *et al., J. Biol. Chem.,* 1947, **168**, 705) provided that the Fe is removed initially. This is effected by passing H₂S through the soln. in the presence of aq. NH₃ (pH 13) and, after removal of the ppt. by centrifugation, expelling the excess of H₂S by evaporation (50° to 60°) under reduced pressure. The folic acid is then determined colorimetrically in an aliquot of the concentrate, after pptn. with ethanol and BaCl₂ if necessary. The accuracy is within $\simeq$ 3%.   W. J. BAKER

1040. Enhancement of ultra-violet absorption of L-ascorbic acid in the presence of D-sorbitol. J. S. Lawendel (Italfarmaco S.p.A., Piazza Coriolana 5, Milan, Italy). *Nature,* 1956, **178**, 873–874.—In the presence of D-sorbitol (0·001 to 7·5 g per 100 ml),

the max. u.v. absorption of L-ascorbic acid is much increased, both in neutral aq. soln. (pH 6) and in acid soln. (pH 2). The absorption curve indicates higher amounts of vitamin C than are actually present. This increased absorption is observed only when a very conc. soln. of the two components is prepared and then successively diluted to the required final concn. and pH. The increase is $\simeq$ 48% above normal value (E₁ᶜᵐ¹% at 265 mμ = 620) for neutral soln. and $\simeq$ 26% above normal value (E₁ᶜᵐ¹% at 245 mμ = 550) for acid soln. The max. increase occurs at a sorbitol to ascorbic acid concn. of $\simeq$ 15:1.   W. J. BAKER

1041. Evaluation of methods and equipment used for sanitation analysis [of flour]. R. H. Cory (General Mills Inc., San Francisco, Calif., U.S.A.). *Cereal Sci.,* 1956, **1** (2), 68–71.—A survey has been made of methods and apparatus used in the U.S.A. for the examination of wheat flour for contamination with insect fragments and rodent filth. It is tentatively suggested that for insect fragments the presence of mineral oil (usually 20 ml for a 50-g sample) during the solubilisation of the flour constituents by acid hydrolysis (enzyme digestion is rarely used) is an advantage, and the staining of undigested vegetable fragments with methylene blue or methyl green helps identification of insect fragments; the use of a Wildman trap during the separation of extraneous matter from digested material gives a better recovery than does the use of a separating funnel. For rodent hairs, staining tends to obscure the hairs and recoveries are better with a separating funnel.   S. C. JOLLY

1042. Factors influencing accuracy in counting insect fragments and rodent hairs [in flour]. R. H. Cory (General Mills Inc., San Francisco, Calif., U.S.A.). *Cereal Sci.,* 1956, **1** (2), 71–74.—The results obtained in a collaborative experiment for the counting of insect fragments and rodent hairs in residues from the digestion of contaminated flour have been examined by an improvised statistical method in order to allocate accuracy ratings to the collaborators. Correlation coeff. have been calculated between number of fragments and hairs reported, time taken and visual acuity, and each factor correlated with the accuracy ratings. A commonly acceptable definition of "reportable" rodent hair is needed. The mean of the number reported in a collaborative analysis is no measure of the true number present, which is probably much higher than the mean.   S. C. JOLLY

See also Abstracts 876, 937, 943, 1064, 1072, 1079.

Sanitation

1043. A method for the simultaneous determination of traces of hydrogen sulphide and carbon disulphide in air. W. Sonnenschein and K. Schäfer (Hauptlab., Spinnfaser A.-G., Kassel-Bettenhausen). *Z. anal. Chem.,* 1956, **153** (2), 107–112.—It is now shown that the results for the determination of H₂S (*cf. Anal. Abstr.,* 1954, **1**, 1246) depend on the volume of air over the liquid. The apparatus and method have been modified to allow for this and to effect other improvements in the procedure.

A. R. ROGERS

1044. The gravimetric determination of potassium in sea water as the potassium tetraphenylboron salt. K. F. Sporek (Plant Protection Ltd., Yalding, Kent,

England). *Analyst*, 1956, **81**, 540–543.—With slight modifications, the method is that previously described (Sporek *et al.*, *Anal. Abstr.*, 1955, **2**, 2339). The sea water (50 g), mixed with conc. HCl in a stoppered flask, is cooled in ice water and treated with a cooled soln. of sodium tetraphenylboron (1% w/v) previously made just alkaline to phenol-phthalein with N NaOH. The potassium tetra-phenylboron ppt. is collected, washed with saturated potassium tetraphenylboron soln., dried at 110° to 120° and weighed. Under the specified conditions no pretreatment of the sample is required. Quoted results indicate satisfactory accuracy. A. O. JONES

1045. The determination of calcium in rain water. I. Davies and G. Hopkinson (Central Electricity Authority, Battersea Generating Station, Kirtling Street, London). *Analyst*, 1956, **81**, 551–553.—The standard method for determining Ca in rain water from atmospheric gauges (B.S. 1747:1951) is time-consuming. The use of EDTA avoids double pptn. of oxalate, but masking of the Cu^{2+} (introduced as an algicide) and evaporation of large vol. of water are necessary. Use of the flame photometer over-comes the need for evaporation, but agreement with the standard method is poor. The agreement was improved when the instrument was calibrated with a soln. simulating London rain water (composi-tion given). For the EDTA method a convenient amount of water is evaporated to $\simeq$ 100 ml and 2 ml of 4 N NaOH, 1 ml of 0·3% KCN soln. and $\simeq$ 0·2 g of murexide indicator are added. The liquid is then titrated with 0·02 N EDTA soln. The results of the examination by each method of 33 samples of rain water are quoted, and the regression equations for predicting the correspond-ing results of the standard method from those of the alternative methods are given. It appears probable that either method gives results for calcium content to an accuracy within the sampling error of the standard collecting gauge.

A. O. JONES

1046. The compleximetric determination of cal-cium and magnesium in waters. R. Červenka and M. Korbová (Inst. of Hygiene, Prague, Czecho-slovakia). *Chem. Listy*, 1956, **50** (2), 306–308.—The successive titration of Ca and Mg with Erio-chrome black T and murexide as indicators is described. J. ZÝKA

1047. Spectrophotometric determination of silica in mineral waters. C. Milani (Inst. Pharmacol. and Exp. Therapy, Pavia Univ., Italy). *Chim. e Ind.*, 1956, **38** (7), 587–590.—Quant. determination of silica in mineral waters is usually made gravi-metrically, no distinction being made between ionic and colloidal forms. By spectrophotometric deter-mination of the known molybdosilicic complex, the ionic portion of silica is measured, and colloidal silica is calculated by difference from the total silica. The absorption spectrum of the molybdosilicic complex and the calibration curve are reported, as well as the experimental results of typical deter-minations on some mineral waters. C. A. FINCH

1048. Problems in estimating fluorides in water. H. P. Kramer, R. C. Kroner and D. G. Ballinger (Robert A. Taft San. Engng Center, Cincinnati, Ohio, U.S.A.). *J. Amer. Wat. Wks Ass.*, 1956, **48** (5), 573–578.—Four variations of the Zr - alizarin procedure for the colorimetric estimation of F in water are compared, *viz*, the Scott - Sanchis,

Lamar and Megregian - Maier photometric pro-cedures as described in "Standard Methods for the Examination of Water, Sewage and Industrial Wastes," 10th Ed., APHA, AWWA and FSIWA, New York, 1955, p. 98, and the recently published Megregian Eriochrome cyanine R method (*Anal. Abstr.*, 1954, **1**, 2676). The effects of interfering substances are examined and distillation techniques are discussed. S.C.I. ABSTR.

1049. The relative stability test [for sewage]. W. L. Tidwell and J. H. Sorrels (Texas Engng Exp. Sta., College Station, Texas, U.S.A.). *Sewage Ind. Wastes*, 1956, **28** (2), 136–139.—The mechanism of the methylene blue stability test for sewage effluents, originally devised by Phelps and Winslow, is ex-amined. It is shown experimentally that relative stability varies indirectly with the amount of org. matter and no. of bacteria and directly with concn. of nitrates, and its relation with dissolved O is shown by a curve which rises to a max. at $\simeq$ 4 to 5 p.p.m. of dissolved O. S.C.I. ABSTR.

1050. Comparative analyses of sewage sludges. M. S. Anderson (Soil & Water Conservation Res. Branch, U.S. Dept. of Agric., Beltsville, Md.). *Sewage Ind. Wastes*, 1956, **28** (2), 132–135.—Various sewage solids and sludges from different stages of treatment were analysed. Tables show (i) a com-parison of the nitrogen and phosphorus contents of activated and digested sludges for 1931–35 and 1951–55, (ii) the min., average and max. contents of Cu, Zn, B, Mn and Mo of activated and digested sludges, and (iii) the origin and percentage com-positions (N, C, P_2O_5, ash, pH) of the sewage sludges studied. The influence of such factors as synthetic detergents, industrial wastes, and the use of home garbage grinders, is discussed. S.C.I. ABSTR.

1051. Chromatographic determination of volatile acids [in sewage]. H. F. Mueller, A. M. Buswell and T. E. Larson (Illinois State Water Survey Div., Urbana, Ill., U.S.A.). *Sewage Ind. Wastes*, 1956, **28** (3), 255–259.—An application for control pur-poses in sewage plants of the method of Bulen *et al.* (*Anal. Chem.*, 1952, **24**, 187) in which org. acids, adsorbed on silicic acid from an aq. solution, are eluted by solutions of varying concn. of n-butanol in $CHCl_3$, is reported. Fairly close agreement was observed between total volatile acidity as deter-mined by direct distillation and the values obtained by chromatographic procedures. J. M. JACOBS

1052. Determination of nitrate in the presence of nitrite in dilute aqueous solution. E. G. White and K. A. Murray (Dept. of Chem. & Chem. Eng., Univ. Witwatersrand, Johannesburg, S. Africa). *J. S. Afr. Chem. Inst.*, 1956, **9** (1), 66–70.—A rapid routine method for determining nitrates in the presence of nitrites in aq. solution (*e.g.*, sewage effluents) con-sists in (*a*) determining nitrites by the Griess - Ilosvay method, (*b*) oxidising the nitrites in a separate portion by low-c.d. electrolysis and deter-mining total nitrates colorimetrically with phenol-sulphonic acid. S.C.I. ABSTR.

1053. The determination of selenium in effluents. D. N. Fogg and N. T. Wilkinson (I.C.I. Ltd., Res. Dept., Alkali Div., Northwich, Ches., England). *Analyst*, 1956, **81**, 525–531.—In the American Public Health Association's tentative method for the determination of Se, differences in the colour of the turbidities produced by pptd. Se in sample and standards render the method unsatisfactory. In

the method now described, the double reducing agent of the A.P.H.A. method (SO$_2$ and hydroxylamine) is replaced by ascorbic acid. The organic matter in the sample is destroyed with HNO$_3$, H$_2$SO$_4$ and HClO$_4$ (the last preventing loss of Se). The residue is diluted with water and distilled with a Br - HBr mixture and H$_2$SO$_4$, the distillate being collected in a flask containing some of the Br - HBr mixture. The same distillation procedure is used with a standard soln. of Se. Aliquots of the standard and sample soln. are diluted and boiled until free from Br and are titrated with N NaOH; aliquots of the standard soln. are then adjusted with HBr to the same acidity as the sample aliquot. The Se in the sample and standard aliquots is then pptd. by addition of ascorbic acid, and after 30 min. the pink turbidities are compared. The accuracy of the method is satisfactory.   A. O. JONES

1054. Determination of volatile oil in effluents. J. G. Sherratt (Public Analyst's Lab., Flag Lane, Warrington, Lancs., England). *Analyst*, 1956, **81**, 518–525.—The apparatus is described and illustrated. The whole of the sample of effluent is placed in a distillation flask, the container is rinsed into the flask with 2·5 ml of acetone, the pH of the liquid is adjusted to 10 with NaOH and distillation is carried out in a current of air through an upward-sloping reflux water-condenser. Attached to the end of the condenser is a short column of active carbon (0·2 g). Ten minutes after drops of acetone have ceased to appear in the condenser the carbon column is detached and eluted with drops of acetone until exactly 2 ml of eluate has been collected. The eluate is diluted to 10 ml with water containing 1 ml of conc. H$_2$SO$_4$ and 1 ml of Teepol per litre, and its turbidity is compared with that of a series of standards prepared by dissolving 1 ml of petrol or other volatile oil in 100 ml of acetone and diluting aliquot portions in the same manner. Modifications of the method to suit certain samples are described and results obtained are discussed.   A. O. JONES

See also Abstracts 854, 870, 875, 882, 895, 896.

Agriculture and Plant Biochemistry

1055. The use of stable diazonium salts as reagents to distinguish flavones and their glucosides. R. Neu (Chem. Forschungslab., Firma Dr. Willmar Schwabe G.m.b.H., Karlsruhe, Germany). *Z. anal. Chem.*, 1956, **153** (3), 183–186.—Diazotised 4-amino-3:6-dimethoxy-4′-nitroazobenzene (Fast black salt K), diazotised 1-amino-4-benzoylamino-2:5-diethoxybenzene (Fast blue salt BB) or tetrazotised o-dianisidine (Fast blue salt B) as 0·1 to 0·5% soln. in 50% aq. methanol give coloured ppt. with 0·1% methanolic soln. of flavones, but not with soln. of their 7-glucosides. The colours are not distinctive, but soln. of the ppt. in dimethylformamide are usually red or blue for 5-hydroxyflavones, with a shift towards yellow for 3-hydroxyflavones.
A. R. ROGERS

1056. Detection and photometric estimation of heptachlorocyclohexane in the presence of hexachlorocyclohexane. V. Šedivec (Inst. for Ind. Hyg. and Occupational Diseases, Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1956, **21** (4), 960–964 (in German).—Heptachlorocyclohexane (**I**) present in commercial hexachlorocyclohexane (**II**) is determined by dehalogenation with Zn, nitration of the resulting chlorobenzene - benzene mixture and

colorimetric determination of the 1-chloro-2:4-dinitrobenzene. To 0·02 to 0·2 g of material in glacial acetic acid (5 to 10 ml) add zinc dust (1 to 2 g), mix, set aside for 15 min., then warm at 60° to 70° for a further 15 min. Cool, and separate the excess of Zn by filtration or centrifuging. To 0·2 ml of the filtrate add nitration mixture (fuming HNO$_3$ - conc. H$_2$SO$_4$, 1 : 2 by vol.) (2 ml) and continue the determination as described by Šedivec (*Anal. Abstr.*, 1957, **4**, 947). The determination is not appreciably affected by octachlorocyclohexane in the original mixture, which must, however, be freed from chlorobenzene by prolonged drying in a vacuum desiccator. The method may also be used for the determination of chlorobenzene in **II** by omission of the appropriate stages; $\simeq$ 0·5% of **I** in **II** can be estimated.
P. S. STROSS

1057. Identification of 3:4-methylenedioxyphenyl synergists by reversed-phase paper chromatography. M. Beroza (U.S. Dept. Agric., Beltsville, Md., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1550–1552.—Insecticide synergists and other compounds containing the 3:4-methylenedioxyphenyl group can be identified in the 2 to 10-μg range by chromatography on paper strips impregnated with liquid paraffin, with 30% acetic acid as the developing medium. The position of the compound on the strip is determined by direct spectrophotometry, and an adapter is used to make measurements at two wavelengths corresponding to a high and low absorbancy value of the synergist. Data for six synergists are listed.   G. P. COOK

1058. Possible interferences in the determination of diethyl p-nitrophenyl thiophosphate (after Averill and Norris) caused by the presence of sulphonamides and their removal by steam-distillation. W. Paulus and H. J. Mallach (Inst. für Gerichtliche Medizin der Univ., Bonn). *Arzneimittel-Forsch.*, 1956, **6** (10), 636–638.—With the exception of Prontosil (2:4-diamino-4′-sulphamylazobenzene), all sulphonamides interfered with the diazo determination of diethyl p-nitrophenyl thiophosphate (parathion). The colour reactions of all diazotised and coupled sulphonamides were studied and absorption curves were drawn for amounts of 1 to 300 μg. By using the Averill and Norris reaction (*Anal. Chem.*, 1948, **20**, 753) and the Bratton and Marshall method (*J. Biol. Chem.*, 1939, **128**, 537), Beer's law is obeyed except by sulphacetamide and phthalylsulphathiazole. The absorption spectra of parathion and all sulphonamides lie too close together for differentiation. The reaction of parathion to steam-distillation was also investigated; it was found to be 85% volatile in neutral solution with no molecular disturbance, while the sulphonamides (except sulphaguanidine) are more volatile in acid or alkaline media.   D. P. FELIX

1059. Chromatography and residue analysis of an organic phosphate insecticide (Demeton). W. J. Magee (Dept. of Zoology & Entomology, Iowa St. Coll., Ames, U.S.A.). *Iowa St. Coll. J. Sci.*, 1956, **30** (3), 411.—Demeton incorporation by bell pepper plants was followed by bioassay, green peach aphid, *Myzud persicae*, and the red-spider mite, *Tetranychus bimaculatus*, being used. The components of the insecticide were separated by reverse-phase chromatography. Phosphate analyses on the separated components showed 20·2% of OO-diethyl O-2-ethylthioethyl thiophosphate (**I**), 54·9% of OS-diethyl O-2-ethylthioethyl thiophosphate (**II**) and 24·1% of

OO-diethyl S-2-ethylthioethyl thiophosphate (**III**). Iodimetric analyses of thiols liberated by alkaline hydrolysis confirmed the values for **II** and **III**. Alkalimetric determination of H_2SO_4 formed by acid hydrolysis of thiono-S gave a similar result for **I**. The various components in plant extracts were separated and determined by similar methods.

S. C. Jolly

See also Abstracts 870, 872, 922, 957.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

1060. A simple robust ultra-micro balance. H. Asbury, R. Belcher and T. S. West (Univ. Birmingham, England). *Mikrochim. Acta*, 1956, (4–6), 598–618 (in English).—The quartz-fibre beam, torsion system and scale-pan cradles were assembled by the method of Wilson and El-Badry. The moving parts are contained in a small air space within an earthed block of aluminium alloy. The balance is robust and stable and records weights up to 700 μg with a precision of approx. 0·01%.

M. F. C. Ladd

1061. Constant-displacement laboratory pump for multiple feeding of test solutions. H. R. Amberg and C. J. Filz. *TAPPI*, 1956, **39** (6), 188A.—A modification of the Brittingham pump is described in which a rubber tube is stretched over four rollers revolving on needle bearings. The rotary action squeezes the tubing, forcing the fluid through it. The pump unit holds 12 tubes, each of which can carry a different solution, and operates at 20 to 200 ml per min.

S. V. Sergeant

1062. Moisture-measuring apparatus [for tobacco]. American Machine & Foundry Co. Brit. Pat. 757,850; Date appl. 13.8.54; U.S.A. 24.9.53.—A continuous stream of shredded tobacco is conveyed on an endless belt past a pair of plate electrodes connected, by shielded conduits, to a dielectric detector (*e.g.*, of the type disclosed in Brit. Pat. 707,238), by means of which the moisture content is determined. The stream of tobacco is then weighed and its moisture content adjusted to a predetermined value.

J. M. Jacobs

1063. A semi-micro extraction apparatus for determination of fatty matter in cotton fibres. F. Franks (Brit. Launderers' Res. Ass., Hill View Gdns., Hendon, London). *J. Text. Inst., Trans.*, 1956, **47** (6), T369–T370.—The apparatus illustrated and described for the extraction of fatty matter from cotton fibre combines the advantages of the Soxhlet extractor with those of the column and tray method (*cf. J. Text. Inst., Trans.*, 1951, **42**, T6). The semi-micro reflux method used gives more accurate results for fatty matter content in the region 0·5 to 1·5 mg than does the column and tray method, and its applicability is limited mainly by the accuracy of the weighing. O. M. Whitton

1064. A new apparatus for nitric acid - sulphuric acid digestion. J. Pien. *Chim. Anal.*, 1956, **38** (10), 361–364.—A full description of the apparatus and procedure for rapid wet digestion of foodstuffs (*cf. Anal. Abstr.*, 1955, **2**, 504) is given. D. G. Forbes

1065. Quantitative paper-chromatography, typical microchemical technique. A. Lacourt (Univ. de Bruxelles, Belgium). *Mikrochim. Acta*, 1956, (4–6), 700–733.—It is shown that, with quantitative paper-chromatography, good reproducibility may be obtained with microgram quantities or even less; *e.g.*, for nucleotides the limiting quantities are between 0·01 and 20 μg. The technique is applicable to all branches of practical and theoretical chemistry. M. F. C. Ladd

1066. A micro-technique for combined analytical or continuous chromatography and electrophoresis. A. Karler, C. L. Brown and P. L. Kirk (Sch. of Criminology, Univ. California, Berkeley, U.S.A.). *Mikrochim. Acta*, 1956, (11), 1585–1592 (in English). —Apparatus is described for carrying out electrophoresis, chromatography or a combination of the two ("electrochromatophoresis") on paper with microgram quantities of material. It can be used for analytical or preparative separations.

A. R. Rogers

1067. Improvements in or relating to viscometers. R. A. P. Wertheim. Brit. Pat. 758,199; Date appl. 13.10.53 and 6.1.54.—A portable, electromagnetic falling-sphere viscometer comprises four coils arranged for connection, in pairs, to form a bridge network. The coil units are spaced apart in axial alignment and a source of electric supply is connected across opposite junctions of the bridge. A detector, *e.g.*, a pair of headphones, is connected across the other pair of junctions. A signal, or change of signal, takes place when the falling sphere passes the coils, thus varying their inductance. J. M. Jacobs

1068. A new viscodilatometer with applications to the Trommsdorf effect in unsaturated polyester resins. M. Gordon and B. M. Grieveson (Tech. Chem. Dept., Roy. Tech. Coll., Glasgow). *J. Polym. Sci.*, 1955, **17** (1), 107–115.—The instrument described combines a normal dilatometer with a plug viscometer and is continuous in operation; only small quantities (amounts not mentioned) of liquid are required. B. J. Walby

1069. Determination of the tensile strength of gels. M. M. Ben-Arie (Sci. Dept., Israeli Ministry of Defence). *J. Polym. Sci.*, 1955, **17** (2), 179–190.— The tensile strength of viscoelastic fluids, gels, etc., can be determined by weighing the amount of gel-column falling under its own weight when extruded from the instrument described. Application of the method to gels containing 2 to 6% of Napalm in petroleum (boiling range 60° to 90°) is described; for this material the tensile strength lies in the range 1·5 to 8 g per sq. cm. B. J. Walby

See also Abstracts 912, 913, 958, 1022.

Optical

1070. Alternating arc interrupter for spectral analysis. J. Mojžíš and L. Háva (Res. Inst. of Electrotech. Physics, Prague, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 320.—The current-impulse generator, constructed for drop-release in polarography, can also be used, after slight adaptation, as an alternating arc interrupter for spectro-chemical analysis. J. Zýka

1071. Effects of finite slit width and stray radiation in differential spectrophotometry. G. F. Lothian (Dept. of Physics, Univ. Exeter, England). *Photoelect. Spectr. Gr. Bull.*, 1956, (9), 207–209.—Graphs obtained by plotting extinction against log concn. are useful because the fractional error in concn. is equal to a length along the horizontal axis. Such graphs are used to illustrate the effects of finite slit width and stray radiation in differential spectrophotometry.   K. A. PROCTOR

1072. Applications of infra-red absorption spectra carried out on small drops of aqueous solutions. C. Duval (Lab. Recherches Micro-analytiques, 11 rue Pierre Curie, Paris). *Mikrochim. Acta*, 1956, (4–6), 741–746.—Infra-red absorption spectra in the range 900 to 1500 cm^{-1} are obtained on a drop of aq. solution confined between small plates of thallium bromoiodide. The anions SO_4^{2-}, HSO_4^-, $S_2O_8^{2-}$, CO_3^{2-}, HCO_3^-, ClO_3^-, ClO_2^-, PO_4^{3-}, HPO_4^{2-} and $H_2PO_4^-$, either singly or mixed, have been identified by this means. The kinetics of the reaction between $K_2S_2O_8$ and KI have been followed on a single drop of solution. The application of this technique to the examination of small drops of blood, milk, wine and chromatographic eluates is suggested.   M. F. C. LADD

1073. Zirconium arc source for infra-red microspectrometer. W. H. Cloud (E. I. du Pont de Nemours & Co., Wilmington, Delaware, U.S.A.). *J. Opt. Soc. Amer.*, 1956, **46** (10), 899–900.—Two optical systems are described for obtaining a magnified image of the crater of a water-cooled 100-watt zirconium arc, the objective being to fill the 6-mm entrance slit-height of the Perkin - Elmer model 112 infra-red spectrometer with microscope attachment. The two systems developed give an increase of incident energy compared with that obtained with a normal 200-watt "globar" source. This increase is approx. 4:1 at 4 μ and 2·25:1 at 14 μ.   B. S. COOPER

1074. New variable-thickness infra-red cell and the infra-red spectra of HF, DF, H₂O and D₂O. R. M. Adams and J. J. Katz (Argonne National Lab., Lemont, Illinois, U.S.A.). *J. Opt. Soc. Amer.*, 1956, **46** (10), 895–898.—The cell described is constructed of Monel metal with silver chloride windows. The cell thickness may be varied continuously from a few microns up to 0·75 mm. The cell has been designed for studies of the i.r. absorption of corrosive liquids such as HF. The spectra of liquid HF, DF, H₂O and D₂O, obtained by the use of this cell, are given, together with a list of absorption coefficients of the principal peaks.   B. S. COOPER

1075. The photo-electric measurement of fluorescence spectra. G. W. King (Dept. of Chemistry, University College, London). *Photoelect. Spectr. Gr. Bull.*, 1956, (9), 227–234.—A single-beam spectrophotometer built round a Müller - Hilger "UVISIR" double quartz monochromator has been used to record the fluorescence spectra of organic fluors such as are commonly used in scintillation counters. The apparatus can also be used to determine the absolute quantum efficiencies of fluorescence and to investigate energy-transfer processes which may occur within the fluorescent material.   K. A. PROCTOR

1076. Cellophane-dye dosimeter for 10⁵ to 10⁷-roentgen range. E. J. Henley and D. Richman (Columbia Univ., New York, N.Y., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1580–1582.—A Du Pont moisture-proof, heat-sealable Cellophane containing a dimethoxydiphenylbisazobis-8-amino-1-naphthol-5:7-disulphonic acid dye was found to be suitable as a dosimeter in the 10⁵ to 10⁷-roentgen range. The irradiation irreversibly decolorises the dye and the transmittance of the Cellophane-dye strip is measured at 655 mμ on a spectrophotometer. A linear relationship between percentage transmittance and gamma irradiation holds up to $\simeq 20\%$ transmittance; at this point the log. of the absorbance is plotted against dose. The 95% confidence limits vary from 60% at 2×10^5 roentgens to 6% at 3×10^6 roentgens. Calibration curves are presented for 0·8-, 2·0- and 3·0-meV beta radiation, and for a ⁶⁰Co gamma source over the dose-rate range of 10⁵ to 10⁷ roentgens per hour.   G. P. COOK

1077. Recording and paper-chromatogram scanning attachments for the SP.500 Unicam. C. E. Kendall and D. G. Lloyd (Dunlop Res. Centre, Birmingham, England). *Photoelect. Spectr. Gr. Bull.*, 1956, (9), 216–226.—An attachment for the Unicam SP.500 is described by means of which single wavelength scans along a chromatogram and the spectra of individual spots can be determined by the same procedure as for normal manual working. A cheap and easily constructed single-beam recording attachment is also described to enable both scans and semi-quantitative spectra to be run automatically. The detailed examination of a paper chromatogram takes about an hour and the conversion of the instrument does not interfere with its use for manual working. The changeover from manual to recording takes only a few seconds.   K. A. PROCTOR

1078. Cell corrections in precision colorimetry. C. V. Banks, P. G. Grimes and R. I. Bystroff (Iowa State Coll., Ames, U.S.A.). *Anal. Chim. Acta*, 1956, **15** (4), 367–372 (in English).—The correction for variations in cell length and absorbance to ensure accuracy in spectrophotometry is discussed theoretically, and three practical methods of correcting for these sources of error are described for unmatched cells. All three methods give the same value for the true reading; one method has been applied to the analysis of rare-earth mixtures (*cf. Brit. Abstr. C*, 1950, 304; *Anal. Abstr.*, 1955, **2**, 2635; **3**, 981).   W. J. BAKER

Thermal

1079. Ubbelohde apparatus for flow and drop points. British Standards Institution (2, Park Street, London). B.S. 894:1956, 16 pp.—This revised standard specifies a metal cup only, and ancillary fittings, for the Ubbelohde apparatus for determining flow and drop points of fats, etc. Spring clips for gripping the cup, which is preferably made of chromium-plated brass, are to be fixed on the case, the tongues specified previously being retained as guides for insertion of the cup. Sleeves can still be used if they are fitted with spring clips. Procedures for filling and using the cup with materials of various types (oil, grease, hard or soft fat) are specified and drawings of the apparatus are given.   W. J. BAKER

See also Abstract 979.

Electrical

1080. Indifferent electrodes. S. K. Chirkov (Ural State Univ.). *Zavod. Lab.*, 1956, **22** (8), 896–898.— New constructions for indifferent, *i.e.*, comparison or standard electrodes, are described. A copper - copper sulphate electrode is prepared by inserting a copper wire, 180 to 190 cm long × 2 to 3 mm in diameter, through a small rubber bung into a glass tube (50 to 60 mm long × 7 to 8 mm in diameter, drawn off to a conical end leaving an orifice 1 to 2 mm in diameter) filled with agar jelly containing $CuSO_4$, prepared by dissolving 25 ml of 10% $CuSO_4.5H_2O$ soln. in 25 ml of hot water containing 1 to 1·5 g of dissolved agar. The tube containing the agar serves as a bridge. The electrode potential at room temp. is + 0·30 V (H scale), which remains practically const. with daily use for two months if, when not in use, it is kept in 5% $CuSO_4.5H_2O$ soln. For an electrode of silver - silver chloride (or other insol. silver salt), the lower part of a longer tube (8 cm) contains KNO_3 or $NaNO_3$ in agar and the upper part contains in addition the insol. silver salt (1 part + 10 parts of agar in 25 ml of a 10% soln. of KNO_3 or $NaNO_3$). A silver wire is inserted into the upper layer. Other types of electrodes can be obtained by the use of wires of Pt, Mo, W and graphite immersed in agar jellies containing quin-hydrone. G. S. SMITH

1081 The potential of the 3·5 N calomel electrode between 40° and 90°. F. Štráfelda and B. Polej (Inst. Anal. Chem., High School of Chem. Technol., Prague, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 185–189.—The potential of a 3·5 N calomel electrode was measured between 40° and 90° at intervals of 5°. The measurements were made by means of a cell consisting of a calomel (3·5 N) electrode and a hydrogen electrode in 0·01 M HCl. During the measurement the temp. of both electrodes was maintained constant. The stated potentials include also the diffusion potentials between 3·5 N HCl and 0·01 M HCl. J. ZÝKA

1082. The standard potential of the silver chloride electrode betwen 60° and 90°. F. Štráfelda (Inst. Anal. Chem., High School of Chem. Technol., Prague, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 190–195.—A hydrogen electrode with a closed automatic hydrogen circuit is proposed. The electrode is suitable for measurements at high temp. With this electrode, the standard potential of the silver chloride electrode between 60° and 90° at intervals of 5°, with an error of 5×10^{-5} V, was measured. The results are expressed by the equation—
$E°_{AgCl/Ag} = 0.19635 - 8.1858 \times 10^{-4} (t - 60) - 3.1144 \times 10^{-6} \times (t - 60)^2 + 7.824 \times 10^{-9} \times (t - 60)^3$, whose validity for the given range was confirmed. J. ZÝKA

1083. Method and apparatus for determining the degree of oxidation of sulphurous gas in the manufacture of sulphuric acid. Soc. Anon. des Manufactures des Glaces et Produits Chimiques de Saint-Gobain, Chauny & Cirey. Brit. Pat. 759,056; Date appl. 8.1.54; France, 8.1.53.—The potential assumed by an electrode immersed partly in the gases, and partly in water in contact with the gases, is measured. This potential is the higher, the lower the proportion of SO_2 in the gases. The gases are passed into a receptacle containing water so that they sweep over the surface of the water, which is constantly renewed to remove any acid formed by part of the gases dissolving in the water. The p.d. between a neutral electrode, *e.g.*, of carbon or platinum, immersed partly in the gases and partly in the water, and a reference electrode (calomel electrode) is measured, *e.g.*, by means of a millivoltmeter of high internal resistance and a potentiometer. J. M. JACOBS

1084. A comparative study of three recently developed polarographs. D. J. Ferrett, G. W. C. Milner, H. I. Shalgosky and L. J. Slee (A.E.R.E., Harwell, Berks., England). *Analyst*, 1956, **81**, 506–512.—The results are recorded and discussed of comparative tests of the merits of the Univector unit of the Cambridge Instrument Co., the Mervyn - Harwell square-wave polarograph, and the single-sweep cathode-ray polarograph of Southern Instruments Ltd. The tests included (*i*) sensitivity for reversible and irreversible reductions at the dropping-mercury electrode, (*ii*) resolution for elements with half-wave potential values very close together, (*iii*) effects of the reduction of a major constituent at a more positive potential on the determination of a minor constituent, and (*iv*) rapidity, reproducibility and usefulness in analytical work. Typical applications to a bronze (Cu, Zn, Ni) and a Mazak alloy (Cu, Pb, Cd, In) are described. A. O. JONES

1085. Polarographic vessel for continuous gas analysis. M. Nedorost (Inst. for Petroleum Res., Brno, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 317–318.—A new, simple vessel for continuous gas analysis, for use in gas chromatography, is described. The usefulness of this vessel in the chromatographic separation of halogens and in the continuous determination of H_2S is indicated. J. ZÝKA

1086. Mechanical drop release in polarography. J. Mojžíš (Research Inst. for Electrotech. Physics, Prague, Czechoslovakia). *Chem. Listy*, 1956, **50** (2), 318–319.—A generator of current impulses was constructed. By means of this apparatus the regulation or synchronisation of drop release in polarography is possible when the two-electrode system is used. J. ZÝKA

1087. The potentiostat as an aid in polarography and amperometry. W. Schwarz (Bundesanstalt für mech. u. chem. Materialprüfung, Berlin-Dahlem, Germany). *Chem.-Ing.-Tech.*, 1956, **28**, 423–427.— In order to maintain a constant potential during polarometric and amperometric measurements the circuits have been expanded to include a potentiostat. The principles and construction of suitable potentiostats are described with the aid of circuit diagrams, and a rigorous mathematical treatment is expounded. Possible applications in the fields of polarography and amperometry are discussed. Further modifications to the devices are being developed and tested. D. F. PHILLIPS

1088. Comparison of different palladium - hydrogen electrodes as pH indicators. J. P. Schwing and L. B. Rogers (Mass. Inst. Technol., Cambridge, U.S.A.). *Anal. Chim. Acta*, 1956, **15** (4), 379–388 (in English).—Trials of different forms of palladium electrode as a pH indicator for single measurements and titrations show that a palladium membrane on which H is generated is the most satisfactory in both aq. soln. and 75% ethanol. A suitable membrane is a 0·013-mm palladium foil coated on both sides with palladium black. A platinum or gold wire (2 cm × 1 mm) on which palladium black has

been deposited is adequate for most purposes, provided it is pretreated with formic acid and is not used in ethanol soln. The effects on the electrode potential of electrode pretreatment, stirring the soln. and presence of O are reported.

<div align="right">W. J. Baker</div>

1089. Ultra-micro potentiometric titrimetric analysis. Differential electrolytic potentiometry in redox systems. E. Bishop. *Mikrochim. Acta*, 1956, (4–6), 619–629 (in English).—Two platinum electrodes are immersed in the titration solution in a platform-type cell, a current of 10^{-9} to 10^{-8} A is passed and the potential set up across the electrodes is measured with a valve voltmeter; the titrant is added from capillary burettes of 0·04 mm bore. Titrations studied show pronounced breaks in potential at the equivalence point, and when using volumes of 0·5 to 1 μl, the accuracy is equal to that of macro-scale work. As little as 6×10^{-10} equiv. can be estimated to within 1%. The method is rapid and eliminates the use of a standard half-cell and salt-bridge.

<div align="right">M. F. C. Ladd</div>

1090. A simplified form of coulometric titration. J. S. Hetman (Ruston & Hornsby Ltd., Lincoln, England). *Analyst*, 1956, **81**, 543–548.—In the method described, an extension of the work of Oelsen *et al.* (*Brit. Abstr. C*, 1952, 249), the current is produced within the apparatus by the use of a zinc electrode system immersed in the test soln. but separated from it by a permeable membrane of pyroxylin and connected through an ammeter to a platinum-gauze electrode also immersed in the soln. The current is regulated by varying the depth of immersion of the zinc electrode. The indicator electrode is of platinum and either a saturated calomel half-cell or a platinum wire coated with AgI serves as reference electrode. Details are given of the application of the method to the determination of Cr and V in alloy steels. The progress of the titration is followed by means of a potentiometer and a sharp end-point is obtained by the use of a Mullard Magic Eye. A. O. Jones

1091. Coulometric titrations with electrolytically plated copper. J. M. Dunham and P. S. Farrington (Dept. of Chemistry, Univ. Calif., Los Angeles, U.S.A.). *Anal. Chem.*, 1956, **28** (10),1510–1514.—Copper metal in contact with cuprous halide solution is a powerful reducing agent, being as effective as Ti^{2+} and ferrous ethylenediaminetetra-acetate. An excess of copper is plated on to a platinum-gauze electrode from cuprous halide solution under an inert atmosphere. The oxidant to be determined is freed of dissolved O and added to the cell. After the reaction is virtually complete, the polarity of the gauze is reversed and the copper is stripped electrically. When all the Cu is removed, Cu^{2+} or I produced in the solution causes a current to flow for end-point determination. The difference between the reduction time and the total oxidation time gives the titration time. The titration cell, amperometric indicator and generator circuits used

were essentially those of Meier *et al.* (*J. Amer. Chem. Soc.*, 1949, **71**, 2340). For titrations of 0·16 to 3·2 mg of Cu^{2+}, the accuracy is within ±0·6%, and for 0·5 to 0·8 mg, ±0·3%; for samples containing 0·4 to 0·8 mg of Fe^{3+}, the accuracy is within 1%.

<div align="right">D. G. Lloyd</div>

1092. Radiochemical analysis by gamma-ray spectrometry. D. H. Peirson (A.E.R.E., Harwell, Berks., England). *Atomics*, 1956, **7** (9), 316–322.—Radiochemical separations, *e.g.*, those normally used for fission-product mixtures or, after irradiation of the sample, in radioactivation analyses, may in favourable cases be avoided by gamma spectrometry on the mixture of radioactive nuclides. A double-crystal scintillation gamma spectrometer is described; spectra are presented to illustrate its potential use in the radioactivation analysis of aluminium for Cu, Ga, Mn and Na, and of minerals for Th and U.

<div align="right">E. N. Jenkins</div>

1093. Improvements in or relating to a method and apparatus for testing the presence of specific atomic elements in a substance. U.K. Atomic Energy Authority (Inventor: J. L. Putman). Brit. Pat. 755,967; Date appl. 21.11.53.—The apparatus, which is more particularly applicable to the assay of inaccessible substances, *e.g.*, in strata traversed by bore-holes, comprises a source of neutrons, a scintillation counter capable of delivering pulses of an amplitude proportional to γ-ray energy (*e.g.*, thallium-activated crystal of NaI) and a pulse-amplitude analyser adapted to identify those output pulses from the scintillation counter corresponding to the γ-radiations from a given (n, γ) reaction.

<div align="right">J. M. Jacobs</div>

1094. A system for the rapid reduction of mass-spectrometric data. B. K. Fritts and C. G. Peattie (Exploration and Production Res. Div., Shell Development Co., Houston, Tex., U.S.A.). *Anal. Chem.*, 1956, **28** (10), 1518–1520.—By using the system described it is possible to reduce spectra of compounds of high mol. wt. six times faster than by normal manual measurements and without operator fatigue. The repeatability of any measurement is equal to that obtained manually, and the data are presented in a form suitable for electronic computing. The system is trouble-free and requires only 1 hr. for the training of an operator.

<div align="right">K. A. Proctor</div>

1095. An apparatus of great accuracy for the determination of methane. B. Domanski, A. Jourdan and C. Eyraud (Lab. Chim. Ind., Fac. Sci., Lyons, France). *Chim. Anal.*, 1956, **38** (9), 322–327.—An apparatus that depends on the resistivity of a platinum filament placed in one arm of a Wheatstone-bridge circuit is described. The gas is introduced into a chamber containing the filament which acts as a catalyst in the combustion of the gas. The accuracy of the method is within ± 5%.

<div align="right">C. A. Slater</div>

See also Abstract 853.

Errata.—December (1956) issue, abstract 3745. Line 13, *for* "50 ml" *read* "5 ml", and *for* "20 ml" *read* "20 μl"; line 14, *for* "2 ml" *read* "2 μl"; line 21, *for* "500 mg" *read* "500 μg". These errata occur in the original paper.

February (1957) issue, abstract 658, line 2. *For* "Okuma" *read* "Omura".

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline	} cryst.
crystallised	
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_{λ}^{t}
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (wt. in wt.)	%(w/w)
per cent. (wt. in vol.)	%(w/v)
per cent. (vol. in vol.)	%(v/v)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_{λ}^{t}
relative band speed	R_{F}
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_{\lambda}^{t}$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\not>$	not less than	$\not<$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, $e.g.$, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^{+}, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^{-}, SO_4^{2-}, PO_4^{3-}, etc., for anions.

ANALYTICAL ABSTRACTS

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